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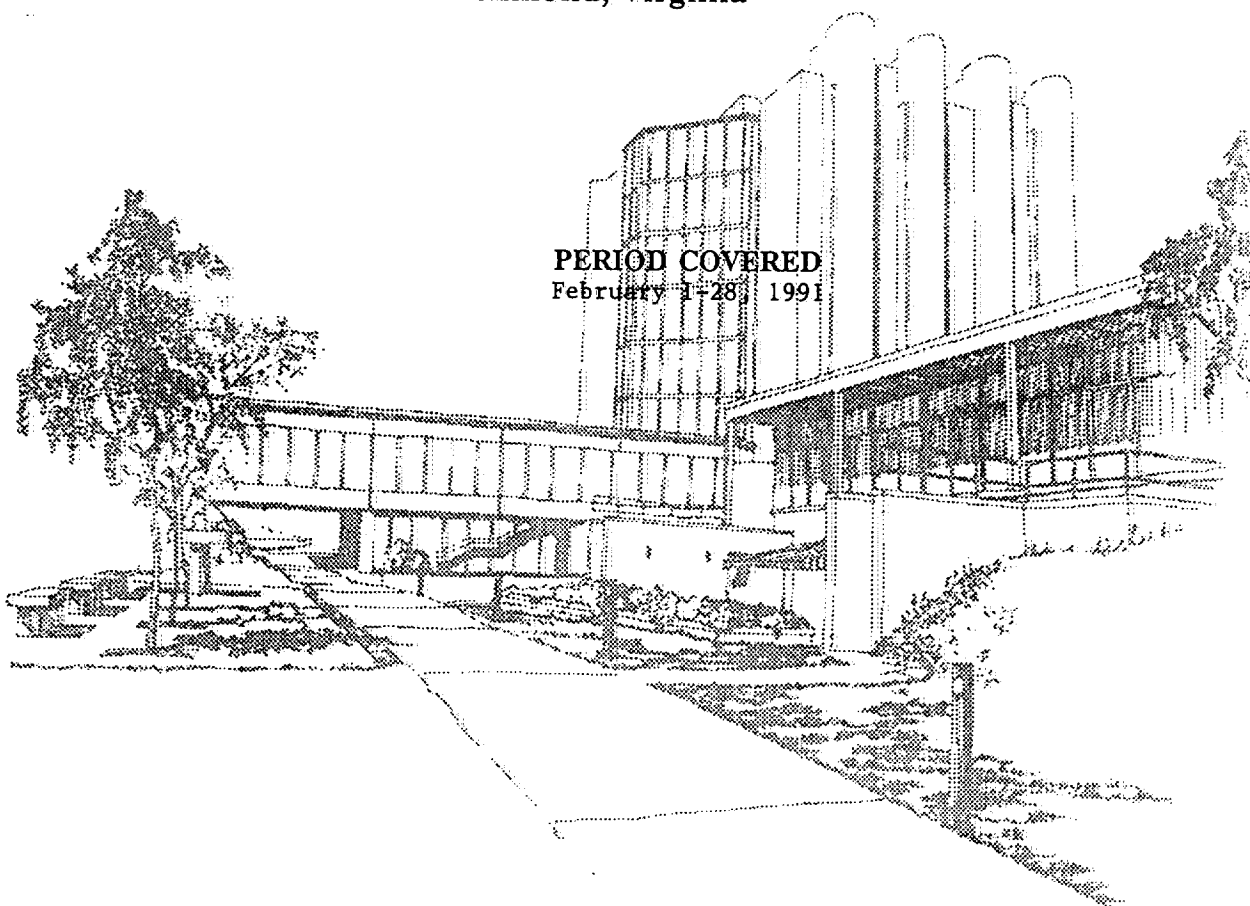
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Research Center
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PERIOD COVERED
February 1-28, 1991



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* N/R = No Report

PROJECT NUMBER : 2108
PROJECT TITLE : New Product and Filter Technology
PROJECT LEADER : W. T. Callahan
PERIOD COVERED : February, 1991

I. FILTER WEB DEVELOPMENT

- A. Objective:** To characterize web-type filter materials and develop subjectively acceptable products with them.
- B. Status:** Filter efficiency of web filters produced with PM web produced on January 3 at James River Neenah Technical Center (JR-NTC) showed removal efficiencies equivalent to the October and November JR-NTC runs and to Tela paper.

PM web samples made on January 28 at JR-NTC were evaluated for rodmaking characteristics. Courtaulds staple web was found to have a shorter capability range, higher rod RTD, and equal variation when compared to Celanese. Celanese material produced with and without crepe showed the uncreped material to have higher rod weight, lower RTD values, and shorter capability range. All samples ran well at medium corrugation but exhibited substantial dusting problems. In general, the Celanese material performed as well as previous Celco-Narrows materials. Filter efficiency data comparing Courtaulds staple web to Celanese staple web showed equal efficiency at same RTD. Efficiencies were similar to previous webs made at James River.

Web samples from February 16, 1991, trials at JR-NTC were evaluated for dusting during rodmaking. Models with insoluble PVOH were unimproved over previous webs. Static electricity in these webs was also substantial. Materials were slit to 5.5 inches and forwarded to American Filtrona for sample filter production.

A Trim KDF2 gamiture was ordered from York to evaluate paper filters at 6mm diameter. Filter making trials with Tela paper at a 6mm filter diameter indicated significantly greater RTD capability than American Filtrona at lower variability. This information will be delivered to AF to drive their process improvement efforts.

II. MACHINERY DEVELOPMENT

- A. Objective:** Develop or modify manufacturing equipment to support Product Development efforts.
- B. Status:**

Strip Application Unit: Adhesive evaluations are ongoing on the Strip Application Unit (SAU). Non-uniformity of adhesive application has required several machine

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adjustments to be made. Group personnel along with Engineering have evaluated the problems and short and longer lead time modifications to the SAU have been suggested.

The application of adhesives in various patterns is being explored as an alternative to the application of paper strips. Quotes have been received from Inta Roto and Jewett Automation to provide glue rollers with patterns.

Coating Equipment: Alliance Machine submitted a quote for design and fabrication of a 12 inch wide coater as specified by PM. A conceptual drawing of Kimberly Clark's proposed new coater was obtained and evaluated for comparison to the PM specifications. This design appears to have fewer features than were originally proposed for the PM coater. The PM specifications were also transmitted to Fox Valley Ltd., the designer/fabricator of the KC machine. This quotation is currently being reviewed versus the Alliance quotation.

The R&D machine shop has completed fabrication of a bracket to hold the Nordson slot nozzle and installation is proceeding.

III. NOVEL FILTERS AND FILTER MATERIALS

- A. **Objective:** To investigate novel filters or filter materials for potential applications where increased efficiency, taste response or a novelty is needed.
- B. **Status:** Sample filters using Tela paper, CA web, and Celanese PM web core materials were obtained from American Filtrona for testing of alternate BOLD 100s designs. Samples using triethyl citrate on tow with Tela core material were also obtained.

PVA and hot melt adhesive samples used by American Filtrona (AF) for concentric filter making were subjectively evaluated by Flavor Technology and the Richmond Panel. The hotmelt was rejected for off-taste and a new hotmelt is being forwarded for trial. Samples were also sent to Analytical Research Division for testing.

48 trays of 98mm paper core concentric filter rods were received from American Filtrona to support additional Product Development testing.

Development of disposable dual Cambridge pad holders for measuring peripheral and core deliveries in concentric filters is ongoing. Die design changes by the vendor have been successful in improving the fit between the different holder components. Further refinements are in progress to improve the smoothness of the interior surfaces of the pad holder.

IV. ALTERNATIVE PLASTICIZERS

- A. **Objective:** Qualify alternative plasticizers which offer a product advantage.

- B. **Status:** A preliminary disclosure was written for consideration of patentability of triethyl citrate plasticizer and the enhanced menthol delivery observed using TEC plasticizer.

V. CARBON STUDIES

- A. **Objective:** Conduct fundamental studies of carbons and carbon filtered cigarettes to better understand their performance.
- B. **Status:** Materials for the production of combined filter rods on a Hauni High-Speed plug-space-plug combiner were ordered and delivered to Hauni Hamburg for a trial during the week of April 22.

A test to verify the mean and variation levels of our existing carbons was designed. The test design provides for the characterizing of carbons on a "standard" sieve screen distribution and for the correlation of PM and Calgon/Pica measurement systems. Resulting information will be used to establish carbon specifications.

A second set of samples was submitted to Flavor Technology for GC headspace analyses. The analyses will determine differences in the amounts of anethole, propylene glycol, and other tobacco components as a function of the filter with/without activated carbon.

Ten drums of SCCW carbon without iron and zinc additives were received from Calgon. Samples were taken from each drum for CCl_4 activity, moisture, sieve size, and hardness measurements.

A TGA method was developed that gives the percent freon adsorbed on dried carbon at 25°C. Envirotrol will measure the CCl_4 activity of the carbon being used to develop a correlation.

Carbon fiber samples were received from Ashland Carbon Fibers and forwarded to Envirotrol for activity determination. Samples include 1/4" staple, mill carbon fiber, and two carbon fiber sheet samples. Filter rods will be produced with the carbon fiber sheet samples.

VI. MENTHOL TECHNOLOGY

- A. **Objective:** To develop alternative mentholation techniques which offer a product advantage.
- B. **Status:** MF Lights were made with Eastman menthol-in-dope filters with/without plasticizer and packed with/without mentholated foil to study menthol stability during aging. Initial smoking data on fresh samples indicate that the models with plasticizer delivered more menthol than the models without PZ. After two weeks aging in desert conditions, models with no plasticizer exhibit less menthol migration from the filter than those with plasticizer. At the same time, the models without plasticizer aged for two weeks in desert conditions delivered equal or more menthol than those

stored in lab conditions. Storage in desert conditions for five days reduced menthol delivery by about 28% for models made with plasticizer and packed in mentholated foil.

MF Lights made with CA and CA/paper filters with two additive levels (4.44% and 7.98% of a PPG/TA/levulinic acid mixture) were subjectively smoked. The CA/paper model with the higher additive level was more similar to the control, but the test model with the lower additive level was judged higher in cooling than the other two models which is consistent with the analytical smoking data. Cigarettes were submitted to Flavor Technology after five weeks of aging in lab conditions.

Two test rolls of aluminum foil with special surface treatment from Reynolds Metals were mentholated at the request of Operation Services. The rolls ran well during menthol application and have been taken to Louisville for evaluation of menthol level and machinability on the packer.

VII. PRODUCT DEVELOPMENT SUPPORT

- A. **Objective:** To provide cigarette/filter design, modelling and testing support for major new product development programs.

- B. **Status:**

Lark: Meetings were held with Kimberly-Clark and Ecusta personnel and plans were developed for future porous combining wrap models.

Half-Nic: The Half-Nic smoking panel completed subjective evaluations of machine made cigarettes with a carbon on tow/CA dual filter. This filtration system was perceived to reduce some of the chemical off taste associated with ART cigarettes, however there was also a reduction in tobacco taste. This filtration system will be re-evaluated once the new totally extracted blend system has been designed by Leaf Department.

Machine made cigarettes have been produced using carbon-on-tow and Bold type filtration systems. We are awaiting CI data. Hand made cigarettes with triple filter designs have been subjectively evaluated. Several models show some promise and further testing is indicated.

VIII. NEW PACKAGING CONCEPTS

- A. **Objective:** Develop or evaluate new packaging concepts which may offer a product advantage.

- B. **Status:** The usage testing by PED was halted to address pack failures incurred during opening. Design changes are in progress and materials are being prepared for further testing.

The ICI Americas co-polymer biodegradable powders used for film packaging were received. Samples of the powders will be sent to the Chemical Research Group for analysis.

PROJECT NUMBER : 2305
PROJECT TITLE : Applied Flavor Investigation
PROJECT LEADER : J. W. Swain
PERIOD COVERED : February, 1991

I. PROCESS SUPPORT

- A. **Objective:** To develop and evaluate process modifications to maintain subjective and physical quality.
- B. **Status:** Analyses of Marlboro control and two test prototype cigarettes with reduced humectant levels in the RL's and RCB have been completed. Subjective evaluations have been initiated to select either the prototype with the target or increased soluble level RL's for POL tests in June.

RLTC trials were completed at Park 500 at three soluble levels to test the original versus the evaporator upgrade system. Analytical and subjective comparisons of the RL's are in progress prior to making Marlboro cigarettes for POL 3646 (Run 3) which is scheduled to be shipped to panelists in April.

Analytical and subjective tests showed no differences between MC DET from trials of CO₂ from the original S1 versus the new S2 CO₂ holding tanks. Qualification of S2 was recommended.

C. **Plans:**

Initiate POL tests of reduced humectant RL's and RCB in Marlboro	April, 1991
Ship POL 3646 (Run 3) Evaporator Upgrade	April, 1991

II. DRY FLAVOR REPLACEMENT

- A. **Objective:** To develop, evaluate and establish specifications for dry flavor replacements.
- B. **Status:** Approvals for the flavor metering equipment at Park 500 have been signed for implementation of the dry flavor replacements in RLB. Scheduling of the preparation and analyses of the solid extract at the Flavor Center are in progress for implementation at Park 500 in May. Analytical and subjective evaluations are in progress on a series of native extracts from an alternate vendor. Samples were submitted to develop analytical specification methods for the flavors at the Flavor Center.

C. Plans:

Implementation at Park 500	May, 1991
Initiate Tests of Replacements in RCB	2nd Quarter, 1991

III. CAST LEAF PROGRAM**A. Objective:** To develop and evaluate flavor systems for cast sheet utilization.**B. Status:** Stepwise substitution of ASTA for RCB will be recommended for the Spanish Marlboro based upon Richmond Panel evaluations of 4% and 7% levels of ASTA. Provisions have been made to supply flavors in April for ASTA production to replace 4% RCB in Marlboro as early as August, 1991.

Cast Leaf Lab trials of SIVA to replace RL's are in progress to evaluate modification in flavors and feedstock prior to further production trials in Cadiz. Since approximately 35 formulations of normal Spanish feedstock in SIVA had failed to produce the flavor and response of the target RL's, changes in the feedstock were tested. Initial subjective evaluations have shown that incorporating at least 20% burley stems in the Spanish feedstock produced attributes in the direction of RL's.

C. Plans:

Complete Feedstock Tests	March, 1991
Select Formulations for Cadiz Trials	March, 1991

IV. NET PROGRAM**A. Objective:** Provide Flavor Technology Support for the New Expanded Tobacco Program.**B. Status:** Subjective evaluations showed normal cased NET from gaseous impregnated #10 Bright to be most similar to cased DET as compared to uncased, precased with sugar only and postcased NET. Analytical data from the trials of 100% NET cigarettes showed differences related to filler weights.

Current strategy involves CO₂ impregnation of filler at Bermuda Hundred and expansion in the 8" Tower. Several NET trials are under analytical and subjective evaluations.

V. OPERATIONS SUPPORT**"PROJECT GRAIN"****A. Objective:** To significantly reduce alcohol levels in PM flavor systems, while maintaining product subjective integrity.

- B. Status: Marlboro cigarettes with a 50% reduction of alcohol in the Burley Top Casing are being made to test on the Marlboro Monadic Panel. POL 0381 is scheduled to be shipped in April. Marlboro cigarettes from large-scale Semiworks trials of further alcohol reductions (75% and 100%) in the BTC have been analyzed and submitted for evaluation by the MC Panel.

Semiworks trials have been requested to evaluate PG rearrangement from the Bright Casing to the AC in efforts to compensate for 25% and 50% alcohol reductions in the Marlboro AC. Lab scale analytical results were promising.

C. Plans:

Ship POL 0381 of BTC Test April, 1991

Evaluate PG Rearrangement March, 1991

PROJECT NUMBER : 2306
PROJECT TITLE : Marlboro Standardization/International Support
PROJECT LEADER : W. R. Bell
PERIOD COVERED : February, 1991

I. MARLBORO STANDARDIZATION

- A. Objective:** Analytical and subjective evaluations of production Marlboro KS/LS.
- B. Status:** The subjectives of January 15, 1991 have been completed and the analytical data is nearing completion. A final report will be issued. Data for the presentations to factory personnel has been compiled. These presentations will begin during the month of March. In conjunction with these presentations with the factory personnel, a time will be discussed for the next Marlboro Standard Run for 1991. Additional factory pickups will be conducted prior to the Standard Run.
- C. Plans:** Present the results of the analytical and subjective data from the Standard VIII Run to the factory personnel and continue with the factory pickups for analytical and subjectives.

II. MARLBORO 83 mm FTB/MEDIUM MILDS/EXTRA LIGHTS

- A. Objective:** To develop a Marlboro line extension in 8-9 mg (Extra Lights) and/or 12-13 mg (Medium/Milds) using cork tipping.
- B. Status:** The initial production of the domestic 83 mm FTB was subjectively monitored by members of Flavor Technology. Marlboro Medium/Milds specifications are set for a 12 mg FTC tar delivery product using standard filler and flavors. POL results and model evaluations indicate the ability to produce a 9 mg FTC tar delivery product with Marlboro subjective character, if desired.

III. DOMESTIC PANEL

- A. Objective:** Provide subjective evaluations (rod aroma and smoking characteristics) for domestic markets.
- B. Status:** The panel has been involved in a three-week review training program; as a result, only three (3) subjective panels were conducted.

IV. INTERNATIONAL SUPPORT

- A. Objective:** Subjective evaluations (rod aroma and smoking characteristics) of cigarette brands in the international market.
- B. Status:** Three (3) panels were conducted during this reporting period.

V. FACTORY SUPPORT

- A. **Objective:** To provide R&D support to the factories regarding process modifications, flavors and finished cigarettes.
- B. **Status:** POL's 0362, 0363 and 3644, using MSA, were made in the Semiworks and have been released. POL's 0364 and 3645 are being remade at Stockton Street the first week of March.

VI. INTERNATIONAL

- A. **Objective:** To provide flavor support to the International Development Group as needed.

B. **Status:**

MF/MF Lights (Japan) Casing and aftercut flavors were provided by Flavor Technology for large-scale primary runs for the evaluation of two blend prototypes.

Project Ring (Korea) Filler was produced February 13, 1991 using L&M Milds leaf blend, Ring A+ aftercut and Gold casing. Cigarettes were made for internal testing.

PROJECT NUMBER: 2307
PROJECT TITLE : Basic Flavor Investigation
PROJECT LEADER : R. W. Hale
PERIOD COVERED : February, 1991

I. ANALYTICAL SUPPORT

A. Objective: To provide analytical support for activities related to development and application of flavoring materials.

B. Results:

1. Samples Analyzed for Project 2305:

Twelve flavor samples were analyzed for flavor revisions/consolidation. The vendors have been notified concerning those samples that did not meet our requirements. One aftercut flavor for "Project Grain" was analyzed.

Samples Analyzed for Project 2306:

One hundred seventy samples of aftercut, casings, filler and cigarettes were analyzed for humectants, sugars, ethanol, water and flavor constituents.

Samples Analyzed for Project 2304:

Three samples of casings were analyzed for the standard components.

Samples analyzed for Project 5001:

Headspace analyses were completed for two samples of packaging material.

II. FLAVOR INVESTIGATION

A. Objective: To develop new basic and applied flavor technology in support of new product development objectives.

B. Results:

1. The identification of components responsible for the off odor in glycerin and triacetin continues. Preparation of known compounds is in progress to aid in the identification of these components.
2. The evaluation of water soluble flavor/cyclodextrins complexes continues.

PROJECT NUMBER : 4016
PROJECT TITLE : Paper Technology
SECTION LEADER : S. D. Baldwin
WRITTEN BY : W. Geiszler
PERIOD COVERED : February, 1991

I. MAGNESIUM CARBONATE PAPERS (G. Bokelman, S. Tafur)

- A. **Objective:** To produce commercial quantities of a magnesium carbonate paper.
- B. **Results:** The evaluation of additional handsheets made by both PM and Ecusta incorporating magnesite and calcium carbonate, with and without 5% magnesium hydroxide, was completed. Sidestream reductions ranged from 38 to 75%. The upper limit for porosity to maintain good sidestream reduction was shown to be .9-10 Coresta. The inclusion of at least 10-12% calcium carbonate is needed to provide acceptable ash appearance. The addition of 5% magnesium hydroxide in the filler system decreases sheet porosity and improves sidestream reduction. Subjective screening of handmade cigarettes suggested that papers at basis weights of 45 g/m² rather than 55 g/m² and those without the addition of magnesium hydroxide were preferred. The sizing combination of potassium citrate, sucrose, and citric acid as developed by Ecusta, however, does seem to improve the magnesium hydroxide-containing papers. It is of interest to note that the exact type of calcium carbonate and magnesium hydroxide did not appear to be critical for paper performance or subjectives. Based on both visibility data and subjective screening results, specifications were established for three papers which were successfully produced in a mill run at Ecusta.
- C. **Plans:** Determine the effects of different paper sizings on sidestream reduction, ash appearance, and subjective performance for the three magnesium carbonate papers.

II. PUFF COUNT CONTROL/ASH FLAKING (B. Geiszler, S. Baldwin)

- A. **Objective:** To evaluate cigarette papers with varying parameters to understand and optimize their effects on puff count, static burn times, and ash flaking.
- B. **Results:** Based on the evaluation of 22 different models prepared for Project BOLD with different papers, three papers were selected for further evaluation in order to make the final selection of the paper for use for a March POL. Three mill runs have been completed by Kimberly-Clark to produce those papers: a 25 g/m² sheet with 30% Albacar chalk at 46 Coresta porosity and 1.7% citrate; a 25 g/m² basis weight sheet with 36% Albacar chalk, 46 Coresta porosity and 1.7% citrate; and a paper with 28.5 g/m² basis weight and 30% Albacar chalk, at the same Coresta and citrate levels. The higher basis weight paper is designed to provide the same flax content as a 25 g/m² paper with 25% chalk.

Papers made with higher chalk content produced cigarettes with reduced puff count at lower citrate levels. Papers with higher basis weight also gave the same result. The

fine particle size chalk also accelerated burn rate. An invention disclosure was filed documenting the finding on particle size, which confirmed earlier work. A product patent may be possible to obtain coverage for the increased basis weight and chalk content papers.

Handsheets were produced to determine the relative impact of various grades of calcium carbonate on handsheet physical properties. The inclusion of Multifex carbonate in handsheets at equal porosity reduces tensile strength relative to Albacar or Omega Fine (0.02 micron particle size). The brightness of Multifex is equivalent to Albacar, and both are higher than Omega Fine. The results for Multifex and Albacar reflect the properties of papers prepared by KC for Project BOLD.

Smoking results for nine Marlboro UL prototypes made with papers containing different ratios of sodium citrate to potassium citrate showed no difference in puff count for sodium/potassium ratios of 0.8, 1.5 and 2.8. This observation held true for all three citrate levels tested (1.0, 1.7 and 2.5%). Eleven Marlboro UL models were made with papers containing different types and levels of calcium carbonate; ash flaking evaluations will be conducted for the models.

- C. **Plans:** Evaluate Project BOLD models for ash flaking and puff count. Evaluate Ecusta papers containing various levels of calcium carbonate for analytical smoking performance on cigarettes.

III. WOOD PULP CIGARETTE PAPERS (B. Geiszler)

- A. **Objective:** To qualify alternative suppliers of wood pulp cigarette papers.
- B. **Results:** Bristol 100 FF and Cambridge KS FF cigarette models were made with Wattens wood pulp papers for qualification testing versus Kimberly-Clark wood pulp grades. Wattens has indicated that they use a mixture of aragonite and calcite (two different crystalline forms of calcium carbonate) in their wood pulp papers. Analytical smoking results for Cambridge and Bristol models made with Wattens wood pulp papers containing aragonite were indistinguishable from the control models made with Kimberly-Clark wood pulp grades, which contain only the calcite form of calcium carbonate. The cigarettes will be evaluated subjectively by the Richmond Panel. Cigarettes were also made with Ecusta wood pulp papers for qualification testing. Analytical smoking is in progress.
- C. **Plans:** Determine analytical and subjective performance of Bristol and Cambridge models made with papers from the three suppliers.

IV. SELECTIVE FILTRATION (B. Geiszler, A. Finley)

- A. **Objective:** Identify smoke components whose delivery correlates with the subjective performance of low sidestream cigarette models.
- B. **Results:** Detailed analyses of mainstream smoke were completed by Analytical Research for fifteen cigarette models with different filter constructions. Statistical analyses of the data are in progress to identify any smoke composition trends. PED subjective assessments of the models show a clear differentiation between models made with a low sidestream paper and those made with a conventional paper. Use of a charcoal filter with the low sidestream paper improves the acceptability rating of the cigarette relative to a conventional CA filter. Treatment of the charcoal with an acid or base does not increase the acceptability.
- C. **Plans:** Correlate analytical smoking results with subjective smoking performance.

PROJECT NUMBER : 4017
PROJECT TITLE : Paper Making Processes
PROJECT LEADER : R. M. Rogers
PERIOD COVERED : February, 1991

I. LOW SIDESTREAM SMOKE PROJECT

- A. **Objective:** Develop proprietary cigarette papers for low sidestream smoke.
- B. **Results:** The papermaking properties of sol-gel derived magnesium compound particles were evaluated in the handsheet lab. The samples exhibit properties typical of sol-gel derived fillers, slow drainage and poor retention with the propensity to restricting porosity. Several conventional papermaking approaches were utilized in an attempt to improve performance (inclusion of retention aids and an increase in water temperature). Both approaches mitigate the negative attributes of these materials. It is recommended that a large sample of this material be prepared and that additional tests be conducted to evaluate slurry properties (contact angle, surface tension). Coating or sizing was also recommended as a possible alternative.

Three additional NY Poly samples containing various levels of hydromagnesite and brucite were evaluated this month (7619-196A, B, & C). Increasing the level of hydromagnesite over brucite improves the papermaking properties of the fillers (porosity/freeness relationship) and sheet opacity.

- C. **Plans:** Continue supporting the production of pilot quantities of cigarette paper at Maine.

II. BANDED PAPERS (TOMORROW)

- A. **Objective:** Incorporate cross directional bands of fiber and/or filler in cigarette papers in order to vary cigarette burn rate.
- B. **Results:** Engineering initiated a secrecy agreement and order with Beloit to design and estimate the cost of constructing a banded paper application device that would be placed above a paper machine's couch roll (a soft rubber covered rotogravure type applicator). Philip Morris' commitment at this time is \$4,000. Construction and evaluation on Beloit's pilot paper machine (only 2 ft. wide but operated at a commercial paper machine speed of 500 ft/min.) is expected to cost in excess of \$67,000 dependent on the number of modifications and runs needed to evaluate this concept. If construction costs are reasonable, the device could be installed on Beloit's pilot paper machine by June 1991.

A trial was conducted at Maine this month to evaluate the rotogravure concept utilizing a modified Daubing Dandy roll. The objective of this trial was to reduce the application rate to $\sim 1 \text{ g/m}^2$ while maintaining band contrast. Although band contrast is hindered by the fibrous nature of the two materials (Cellulon or Buckeye Cellulose), due to separation of the fiber and water mix, the materials transfer to the

base sheet. Homogenizing these materials should improve the flow/separation character of the slurries. These trials underscore the necessity of placing the rotogravure device above a vacuum to assist in transfer. The drum must contact the sheet and the alignment of the application device with the base web is critical. Maintaining a clean drum may also be a problem. These factors will be included in the design criteria for the unit that is being constructed at Beloit.

- C. **Plans:** Repeat trials at Maine utilizing homogenized Cellulon to improve band contrast.

III. PROPRIETARY FILTER MATERIAL

- A. **Objective:** Develop a proprietary filter material in web form.

- B. **Results:** Two trials were conducted at James River's Neenah Technical Center this month. The objective of the first trial was to evaluate the web making characteristics of CA obtained from various sources (Courtaulds and Celanese) for scale-up to a mill run in late March 1991. Celanese CA staple (round CA) dispersed better and required less refining to form an acceptable web. Courtaulds CA required four times the refining to achieve a comparable web. The method of dispersion (low consistency) also improves the properties of the web.

The second trial was conducted to evaluate the inclusion of polyvinyl alcohol (PVOH). The objective of this trial was to improve PM web's tensile strength while reducing the tendency to lint during filter making. A portion of the web rolls will be sizing with soluble PVOH. Additional rolls were produced with an insoluble form of PVOH (insoluble in water at <150°F) added as a filler to the sheet (2.5% and 5%). It was anticipated that once the sheet contacted the Yankee, the PVOH would solubilize and migrate throughout the web during the drying process. Manipulating the moisture level of the web contacting the Yankee and the drying temperature resulted in a modest improvement in the sheet's tensile strength. Dye testing indicates that most of the PVOH particles are only partially dispersed. This approach would require additional development efforts to optimize the PVOH particle size, response to temperature or moisture and the time, temperature, and moisture balance on any paper machine's dryer.

In its current configuration (75% cut CA and 25% refined softwood, no PHOH addition or calendering) PM web produced at James River's Gouverneur mill should approach practically all the properties of Tela web. At the same sheet weight it is projected that MD tensile will be -0.9+ kg/in versus 1.3 kg/in for Tela web. Both porosity and caliper will be slightly greater than Tela web (5500 Coresta and 6.0 mils versus 3600 Coresta and 5.0 mils respectively). Given acceptable formation on the paper machine wire, web properties are highly contingent on the amount of disruption caused by the action of the creping blade. Every effort will be made to minimize this disruption while maintaining acceptable crepe quality and elongation properties. Off machine calendering is also being investigated as a possible option to reduce linting.

One large roll of 100% softwood was also produced as a duplicate of Tela web. The sheet is slightly more porous, 5300 Coresta versus 3600 target. The James River representative believes that Tela web can be duplicated at the Gouverneur mill location.

C. Plans: Complete a mill trial of PM web at James River's Gouverneur mill.

Evaluate off machine calendering as a possible method to reduce web linting.

PROJECT NUMBER: 4018
PROJECT TITLE : Paper Product Development
PROJECT LEADER : B. L. Goodman
PERIOD COVERED : February, 1991

I. REDUCED SIDESTREAM CIGARETTES (B. Goodman, B. Floyd)

A. Objective: Develop subjectively acceptable cigarettes with reduced sidestream.

B. Results:

Single Wrap Superslims: CI data for the January test run of single wrapped Superslims showed all physical specifications to be close to targets, and mainstream tar deliveries varied according to the base Coresta values of the papers. The sidestream panel smoked models made with papers at each end of the permeability specification range (11.5 \pm 2 Coresta), each at 10.4% monopotassium phosphate. The majority of the panel could not tell the difference between the cigarettes. The control double wrap and the target Coresta model have been forwarded to Chemical Research for room studies.

Cigarettes from a menthol machinability run in February have been tested for sidestream visibility and ash appearance. Bobbins for this run were selected at the middle permeability level of 11-12 Coresta, and several bobbins were rewound with the wire side out. Neither test model showed any problem with staining behind the charline, and no difference in ash appearance could be seen due to the difference in direction of the paper. Sidestream visibility was reduced by 77% for the control, and 69% and 73% for the two test models. Another set of cigarettes with non-menthol filler has been run in Louisville, and these models will be tested to see if there is a real difference in sidestream visibility between the two paper directions.

A set of QA standards was prepared by Kimberly-Clark with different levels of monopotassium phosphate and calcium carbonate. Testing of the sheets will be coordinated by Analytical Research, and one additional sample with a higher level of chalk will be added when Kimberly-Clark runs their next production run.

Lotus: Low sidestream paper (V1 - 47 g/m², 33% CaCO₃) was coated with two levels of potassium phytate from Chemical Research. A control was also coated with monopotassium phosphate at an equal potassium level. Cigarettes have been made with these papers and are awaiting evaluation.

Ambrosia II: The 47 g/m² low sidestream paper (V2) at 4.5 Coresta was coated on the sizepress with monopotassium phosphate to give 12% on the paper. Part of the bobbin was also coated with Aromatek-245 at the same level as in last years POL tests. The finished bobbins were given to Product Development-USA for making cigarettes.

Conventional and low sidestream papers were coated for a room odor study to be conducted by PED with cigarettes of varying degrees of sidestream visibility

reduction. Different combinations of monopotassium phosphate, malonic acid and Aromatek-245 were applied with the sizepress and tunnel dryer systems. Cigarettes have been made and are awaiting sidestream visibility before turning them over to Chemical Research and PED.

- C. Plans: Complete the visibility testing of single wrap Superslims, and assist in the evaluation of paper additives and Coresta levels on the next millrun by K-C.

Evaluate cigarettes with potassium phytate coating on the paper, and coat the 47 g/m^2 paper with a non-volatile acid in combination with monopotassium phosphate.

Coat the new magnesium carbonate papers from Ecusta with various additives, and make machine made cigarettes for evaluation.

II. SIDESTREAM VISIBILITY TESTING

- A. Objective: Determine sidestream visibility of experimental cigarettes.

- B. Results: Seventeen models with varying levels of monopotassium phosphate on 47 g/m^2 base papers V1, V2, and V3 from the Ambrosia II project were tested on the 8-port. Results have been described in a memo. Generally, the visibility followed the expected trends for additive level and Coresta permeability.

A number of handmade cigarettes were constructed from handsheets from Chemical Research and with magnesite papers, both for visibility testing and for subjective screening.

- C. Plans: Continue testing for sidestream visibility of handmade cigarettes on the single port instrument.

Determine 8-port visibility for machine-made experimental cigarettes.

PROJECT NUMBER : 4022
PROJECT TITLE : Product Development, Affiliates and Licensees
PROJECT LEADER : R. E. Tinker
PERIOD COVERED : February, 1991

I. PROJECT GOLFO (El Salvador)

- A. **Objective:** Develop an acceptable L&M 100's Regular for El Salvador.
- B. **Status:** C.I.'s are complete and cigarettes have been submitted to the Richmond Panel for subjective approval.
- C. **Plans:** If approved, El Salvador will apply for CPC approval. El Salvador plans to launch this line extension in 2nd Quarter, 1991.

II. L&M L.S. PROJECT (Dominican Republic)

- A. **Objective:** Develop an acceptable L&M L.S. Box product for the Dominican Republic market.
- B. **Status:** First set of prototypes were rejected by the Leaf Panel and International Product Development personnel. Prototypes have been remade and submitted for C.I.'s.
- C. **Plans:** If C.I. results are on target, samples will be submitted to the Leaf and Richmond Panels for subjective approval.

III. MARLBORO ASTA TRIALS (Spain)

- A. **Objective:** Evaluate Asta sheet material in the Spanish Marlboro to replace RCB.
- B. **Status:** Asta vs. RCB survivability tests are complete. Marlboro cigarettes with 4% and 7% Asta sheet have been evaluated by Richmond Panel versus a control.
- C. **Plans:** Subjective results by Richmond Panel indicate a blend change is acceptable. The Richmond Panel recommends that if a blend change is implemented, that it be done in two stages of inclusion as per samples evaluated.

PROJECT NUMBER : 4030
PROJECT TITLE : Product Development, Export
SECTION LEADER : J. N. Smith
WRITTEN BY : R. S. Slagle
PERIOD COVERED : February, 1991

I. Marlboro Program (Japan)

- A. Objective:** Replace unavailable recon grades in the Marlboro Japan blend through blend reformulation.
- B. Status:** Results were received for the Danchi III Marlboro/Marlboro Lights blend test. The cigarettes included in this test were produced in November, 1991 at JT's Odawara factory. The test blend used was the 55% offshore/45% domestic tobacco blend that is scheduled for commercial production on March 4, 1991. The following models were evaluated:

Marlboro KS Control (Cork Tipping)
Marlboro KS Test Blend (Cork Tipping)
Marlboro Lights KS Control (White Tipping)
Marlboro Lights KS Test Blend (White Tipping)

All models were rated significantly different from each other in strength.

For both combined and individual smoker groups, there was no significant difference in liking for the Marlboro Red KS test and control models. In overall liking, the Marlboro Lights KS test and control models were not found to be significantly different from each other. The average liking ratings for Marlboro Lights KS were significantly higher than the Marlboro Red KS scores.

- C. Plans:** Evaluate three blends for Marlboro Japan, in an attempt to return the blend to 50% domestic/50% offshore:
1. 55% offshore/45% domestic (variation of new production blend)
 2. 60% offshore/40% domestic (with 6% DETA inclusion)
 3. 50% offshore/50% domestic

II. Price Value (Korea)

- A. Objective:** To develop a viable Price Value Entry for the Korean market.

- B. **Status:** Results from SCP test E-L978 have been received. Models evaluated in this test series were:

L&M Milds @ 8.0 mg tar
Mild Seven Lights
L&M Milds @ 12 mg tar
PM Lights

For combined smoker groups, there was no significant difference in liking among the four prototypes. Within individual smoker groups, 88 Lights smokers rated L&M Milds @ 8 mg tar significantly higher in liking than L&M Milds @ 12 mg tar. 88 Deluxe Milds smokers gave L&M Milds @ 8 mg tar significantly higher liking scores than those for Mild Seven Lights, and L&M Milds @ 12 mg tar received significantly higher liking scores than Mild Seven Lights among Pine Tree smokers.

- C. **Plans:** Prepare for launch of a Price Value Entry in Korea, currently designated as a contingency for 1991.

PROJECT NUMBER : 2304/4031
PROJECT TITLE : Product Development, U.S.A.
SECTION LEADER : C. B. Altizer
WRITTEN BY : G. N. Yatrakis and J. L. Spruill
PERIOD COVERED : February, 1991

I. LOW TAR/HIGH FLAVOR

PROJECT BOLD

- A. **Objective:** Develop a 1mg 85mm and 2mg 100mm regular and menthol product competitive with Now and Carlton.
- B. **Status:** Primary is complete for improved flavor models using optimized paper with TELA and PM web filters.

Forty-eight trays of 98mm paper core concentric filter rods were received from American Filtrona. Sampling and testing are in progress.

Three mill runs were completed by Kimberly-Clark to produce papers for Project BOLD: a 25 g/m² sheet with 30% Albacar chalk at 46 Coresta porosity and 1.7% citrate; a 26 g/m² basis weight sheet with 36% Albacar chalk, 46 Coresta porosity and 1.7% citrate; and paper with 28.5 g/m² basis weight and 30% Albacar chalk, at the same Coresta and citrate levels.

Laboratory work is continuing to develop new flavor systems that enhance smoothness while maintaining impact.

- C. **Plans:** Analyze data and prepare for March POL testing.

II. PROJECT AMBROSIA

AMBROSIA I

- A. **Objective:** Develop a 23.0 circumference aromatic sidestream product as well as a 24.8 circumference 85mm and a 24.0 circumference 100mm and apply this technology to other products.
- B. **Status:** Adhesive stability tests of two 10-gallon batches of adhesive incorporating Aromatek-150 at a 5% wt/wt target are in progress at National Starch. The result of these stability tests will determine which batch of Aromatek-150 we ask FD&O/Givaudan to duplicate. Two POL's incorporating Aromatek-150 coated on paper have been requested (0576 and 0667). Cigarette paper for these POL's will be requested for paper coating at Ecusta.
- C. **Plans:** Produce product with new production batch and finalize specifications.

AMBROSIA II

- A. **Objective:** Develop K.S. and 100mm low smoke, low odor and low smoke/low odor prototypes.
- B. **Status:** Requests for cigarette making, using LSS, LSS/A-245, and charcoal filters for duplication of the original POL's have been submitted. Paper analysis is complete, and the charcoal filters are in-house.

Blend component samples with Regular cigarette paper and LSS cigarette paper were made on February 25. These samples will be submitted for visibility testing and room aroma (PED) evaluation.

Conventional and low sidestream papers were coated for a room odor study to be conducted by PED with cigarettes of varying degrees of sidestream visibility reduction. Different combinations of monopotassium phosphate, malonic acid, and Aromatek-245 were applied with the size press and tunnel dryer systems.

- C. **Plans:** Continue model making for improvements.

III. PROJECT MARLBORO

- A. **Objective:** Design and develop Marlboro 83mm FTB products as possible line extensions.
- B. **Status:** Initial test market allocation quantities of 83mm FTB were completed on February 20, 1991, at Stockton Street.

A factory trial of MF Medium KS SP was conducted at the M/C on February 26, 1991, to evaluate packaging alignment and to finalized tipping paper pressure drop. Cigarettes were submitted to CI on the same date. A factory trial on finalized packaging is expected the week of March 11, 1991.

- C. **Plans:** Monitor test market.

IV. BRAND EXTENSIONS

- A. **Objective:** Design and develop extensions and modifications to existing brand families.
- B. **Status:** POL tests are scheduled to evaluate the B&H menthol, Marlboro menthol and an "N"-type blend and flavor system in an 83mm full flavor and lights configuration. Prototypes have been made and analyzed to determine optimum menthol application to achieve the targeted menthol-per-puff as specified by PED. Production of the first two POL's (FF and Lights with B&H menthol blend) for shipment in mid-March should be complete by March 1, 1991.

PED has requested B&H Menthol KS models in white and cork tipping and B&H Lights Menthol KS models in white and cork tipping for extended internal smoking evaluations. Semiworks requests have been submitted.

- C. Plans: Complete POL production and develop models for additional consumer testing.

PROJECT NUMBER : 5001
PROJECT TITLE : Packaging Studies
PROJECT LEADER : B. Mait
PERIOD COVERED : February, 1991

PACKAGING STUDIES

A. Objective: Provide technical packaging support to Manufacturing, Manufacturing Services, Engineering, Purchasing and Quality Assurance. In addition, assist New Products Directorate in evaluating new packaging concepts and products.

B. Results:

Supplier Reviews

Meetings were held with Eastex Inks (water-based ink manufacturer) and Pierce & Stevens (water-based lacquer manufacturer) to discuss chemical formulation requirements and analytical procedures used to qualify these products for use in packaging materials.

A meeting with Mobil Chemical Company was held to discuss improved barrier properties for water and organics on their package film.

Innovation Center

Members of the Project Gold team presented the timetable for the April 20 factory trial of the pre-applied adhesive in Cabarrus.

PROJECT NUMBER : 1309
PROJECT TITLE : Cast Leaf Development
PROJECT LEADER : G. Gellatly
PERIOD COVERED : February, 1991

CAST LEAF DEVELOPMENT

- A. Objective:** Develop subjectively and physically acceptable reconstituted tobacco sheets for domestic and international application using cast leaf technology and proprietary binder systems.
- B. Results:** **Cast Leaf Pilot Plant** - Purchase orders were issued to Berndorf for the primary and secondary sheet dryers and to Williams for the tobacco feedstock grinder. The P&ID and GA drawings were finalized. Demolition work in C Pilot Plant is complete. The specification for the reverse roll coater has been prepared and an order will be placed in March. Pilot plant construction is now expected to be complete in October.

Cast Leaf Development - Cast leaf formulations continue to be subjectively evaluated and compared with existing recons and their combinations in 100 percent recon cigarettes and 24 percent blends with Marlboro master blend (burley/bright/ oriental strip). Small scale machine made cigarettes of these 24 percent blends have been made to quantify the effect of formulations on tar deliveries, static burn, etc.

Sheet quality has been shown to be effected by the type of mixer used for slurry making. The characteristics of each mixer (Ross, Waring, and Fitzmill) will be quantified to select mixers which might be evaluated in the Cast Leaf Pilot Plant.

Cadiz - The development of SIVA flavor systems for future Cadiz trials is continuing. An increase of burley stem content in the tobacco blend is being evaluated to increase the subjective impact of SIVA. Quest flavor #4 is expected to be delivered in March for subjective evaluation. This Quest #4 was formulated to overcome the physical separation observed with Quest #3. Cadiz trials are not expected to be run in March as the Versator deaerator has not yet been shipped from U.S.A.

C. Plans:

1. Determine the conditions of OV, packout temperature, and packing density necessary to induce ASTA product darkening in the laboratory.
2. Rerun temperature and chemical profiles in packing cases in Cadiz during summer conditions.
3. Continue binder and flavor development work to optimize the cast sheet formulation for USA and TSA.
4. Develop a trial schedule for SIVA trials in Cadiz the week of April 8, 1991. These trials will also evaluate the effect of the gum eductor and air removal from slurry on sheet quality.

5. Continue work with PM Engineering on design and installation of the Cast Leaf Pilot Plant.

PROJECT NUMBER : 1503
PROJECT TITLE : Modified Smoking Materials
PROJECT LEADER : W. A. Nichols
PERIOD COVERED : February, 1991

I. SIDESEAM ADHESIVE METERING SYSTEM

A. **Objective:** Evaluate a sideseam adhesive metering system for use in the Semiworks and as a prototype for Production.

B. **Results:** To determine if installation of a sideseam monitoring system would increase cigarette maker efficiency, historical data has been gathered on cigarette maker downtime attributed to adhesive application. Data specific to adhesive defects during rod formation is unavailable but significant losses in productivity do occur due to rod breaks, of which, adhesives are a cause. Arrangements will be made to monitor makers in the factory and define the reasons for rod breaks.

The sideseam adhesive system was installed in the Semiworks. Cigarettes were produced with three lapseam adhesives at flowrates from 0.6 to 3.8 cc/1000 cigarettes. Lapseam quality and subjective testing is being conducted on the samples. The sideseam monitor will initially be used to collect data on typical adhesive flow variation.

C. **Plans:** The optimum adhesive application rate will be established for currently used lapseam adhesives.

II. STEMMERY WASTE

A. **Objective:** Determine the feasibility of reclaiming tobacco from stemmery waste.

B. **Results:** Stemmery waste contains approximately 25 percent tobacco particles. Waste samples from various bright stalk positions were submitted to PEMM-CORP for sand and tobacco separation. Initial analysis suggests that wet separation techniques are superior to pneumatic methods.

C. **Plans:** Economic and environmental studies will be conducted.

PROJECT NUMBER: 1806
PROJECT TITLE : New Tobacco Processes
PROJECT LEADER : T. C. Holland
PERIOD COVERED : February, 1991

I. PRIMARY IMPROVEMENT PROGRAM

- A. **Objective:** Characterize the existing primary process to establish baseline thermal history, chemical changes, and flavor reactions. Identify equipment and process modifications to simplify the primary process to provide a low operating cost and highly flexible primary operation.
- B. **Status: Thermal Treatment of Burley** - Modification of the Sargent tray dryer is complete and checkout of the unit is in progress. The tray dryer is designed to provide controlled drying conditions of cased burley tobacco to establish the chemical and subjective impact of different thermal treatments.

Samples for subjective and chemical analysis will be generated in March with analytical and subjective correlation completed by late May.

Flow Thru Hopper Trials - An economic analysis was conducted by Operations Analysis to establish estimates of dollar savings available from improved yields and reductions in tobacco weight projected for the cut filler delivery program. An annual savings of \$220,000 per billion cigarettes produced would be generated by achieving the projected goal of the cutfiller delivery program of 40 mg weight reduction and a 1.0 percent yield improvement.

A job order was prepared for the purchase of the Rothmans Flow-Thru Hopper for the Mark 9 maker. The unit will be evaluated for improved cutfiller survivability through the maker with both conventional and non-pneumatic cutfiller delivery.

A meeting was held with representatives from Rothmans to outline the joint development of a cutfiller delivery system. The system will include non-pneumatic cutfiller delivery to the maker, external maker winnowing, and modifications to the Protos hopper. A proposal from Rothmans is expected in early March.

Improved Stem Utilization Trials - A study has been initiated to evaluate improved utilization of DIET stem (removed from the VT separator) by shredding or cutting to an acceptable product that can be reintroduced directly into the blend. The DIET stem is currently used as a feedstock for RL.

DIET stems were shredded at a series of moisture contents using a Sprout Bauer refiner. Samples were collected for CV, sieve and objectionable stem content. Preliminary results will be available in early March. The study will be extended to include treatment of winnowers.

- C. **Plans:** A capital appropriation will be prepared to provide funding for the joint development of a non-pneumatic cutfiller delivery system.

II. CARDWELL REORDERING CONVEYOR

- A. Objective: Develop improved DIET reordering system.
- B. Status: A six deck Cardwell vibrating conveyor has been purchased for evaluation as a second stage reordering conveyor. Evaluation of both spray reordering and humid air reordering can be accomplished with the unit. Delivery of the unit is expected at the end of March.

In cooperation with Dr. J. Crump and W. Winterson, a second series of reordering tests was conducted to establish the conditions required for humid air reordering of DIET. The air conditions for the Cardwell reordering unit have been specified. A cost estimate for the air handling equipment is being prepared.

- C. Plans: Layouts for the installation of the Cardwell in D Pilot Plant will be prepared.

III. RF TREATMENT OF IMPREGNATED FILLER

- A. Objective: Investigate the use of Radio Frequency treatment of impregnated filler to improve the DIET product quality.
- B. Status: Test results indicate that RF drying does not provide adequate treatment for expansion of either liquid or gaseous impregnated filler. The unit will be returned to the vendor at the end of the month.
- C. Plans: No further studies of RF treatment of impregnated filler are planned.

IV. BL PLANT ENVIRONMENTAL STUDY

- A. Objective: Identify the point source stack emissions and waste water effluents at the BL Plant. Establish methods for reducing emissions.
- B. Status: Material balance sampling around the sheet dryers was conducted to confirm the findings of the air emission study. Process and water sampling was also conducted to isolate and quantify water effluent from the BL Plant. All test results are expected in early March.
- C. Plans: Process changes at the BL Plant required to reduce air and water emissions will be investigated.

PROJECT NUMBER : 1810
PROJECT TITLE : ART Process Development
PROJECT LEADER : D. R. Fox
PERIOD COVERED : February, 1991

I. LIQUID ABSORBER PROCESS DEVELOPMENT

- A. **Objective:** To develop a second-generation supercritical extraction process for nicotine using a liquid absorber rather than stems.
- B. **Results:** M. W. Kellogg is soliciting cost estimates on the liquid absorber design, which is the final phase of the project. All pilot plant work on the liquid absorber has been completed, so the extraction process has been shut down.
- C. **Plans:** Kellogg's final report on the design project is expected in March.

II. LIQUID ABSORBENT TREATMENT PROCESS DEVELOPMENT

- A. **Objective:** To develop separation, utilization, and disposal processes for the liquid absorbent effluent from the second-generation ART extraction process.
- B. **Results:** Bench-scale aerobic and anaerobic systems for treating the liquid absorber effluent continue to operate. The aerobic system has been running with high dilution (50:1) and residence time (8 days) initially, and has produced effluent with a COD of 50 mg/l and no detectable nicotine. As the testing proceeds, less dilution and shorter residence times will be evaluated.

The anaerobic system has been more difficult to start, since oxygen enters the seed during transport and startup, thus inhibiting the methane-forming microorganisms. This system is now being fed intermittently to all the methane-formers to recover.

Development Engineering has completed a design for the scale-up of the activated sludge system. Construction of the aeration vessel and clarifier will begin in March.

Electrosynthesis, a firm contracted to perform exploratory research on electrochemical techniques of handling liquid absorber effluent, reports that nearly 100 percent of the nicotine can be destroyed, but high potential electrodes are required. They are sending a sample of the treated effluent for our analysis to determine the products of the treatment.

- C. **Plans:** Continue process development of the various effluent treatment processes.

III. BL PLANT WATER TREATMENT

- A. **Objective:** Identify and develop techniques for reducing levels of targetted constituents (e.g., nitrates and phosphates) in the discharge water from the BL Plant.

- B. **Results:** Analysis was completed on samples taken from the effluent of the TME, Bauer extractor, and Vetter press on the stem washing process. Filtration indicates that solids, including both tobacco particles and insoluble salts, accounts for about 50 percent of the phosphate loading in these streams, as well as significant effects on COD/BOD. However, filtration had no effect on nitrate and ammonia levels.

The stem washing operation is carried out in a semi-continuous mode, with the process started up and shut down each shift. Samples taken over time indicate that considerable changes occur in total phosphorous and solids concentrations as the process runs. This and other results suggest that it may be possible to alter extraction conditions to improve the selectivity for nitrate reduction.

- C. **Plans:** Tests are planned to determine the size distribution of the solids to determine what type of filtration equipment would be required. Alternatives will also be evaluated for nitrate and ammonia reduction.

PROJECT NUMBER : 1811
PROJECT TITLE : Process Chemistry Development
PROJECT LEADER : G. D. Keritsis
PERIOD COVERED : February, 1991

I. CAST LEAF DEVELOPMENT

- A. **Objective:** To investigate appropriate binder systems to be used as an alternative to the current processes.
- B. **Results:** The physical testing of various reconstituted tobacco sheets made in the Cast Leaf Lab has been completed. The data indicates that guar binder produces much stronger sheets than pectin or pectin/guar binders, and that the preliminary subjective screening with handmade cigarettes did not show any significant advantage of pectin over guar. Both binders were found to be subjectively acceptable. Additional sheet quantities with 8 parts guar and varying levels of DAP (0, 1.5 and 2.5) were made in the Cast Leaf Lab for machine made cigarettes and further testing (CI, subjectives).

A method that improves the processability of a "hybrid" BL/RL sheet in the laboratory has been developed, and the plans are to scale-up the procedure in the Cast Leaf Lab during the week of 3/4 for further testing (physicals/subjectives). The method involves the treatment of a portion of the tobacco feedstock (20-40 percent) with DAP/ NH_4OH as per RCB followed by an acidification step prior to adding the binder with the balance of the tobacco feedstock to form a combined slurry for casting.

Several SIVA formulations were made in the Cast Leaf Lab in an attempt to match the RLB/RLTC (50/50) subjectives for TSA. The subjective screening of these formulations is currently in progress by Flavor Technology personnel with handmade cigarettes. Attempts to improve the SIVA sheet physicals by increasing the guar binder level from 4 to 6 parts in the formulation containing a 50/50 mixture of 120 and 400 mesh size TSA feedstock were not successful. This tobacco blend was found to require 10 parts guar to produce a very good sheet, free of cracks. The problems may be due to the tobacco particle size distribution in the blend. This problem is currently being investigated.

Other binder systems currently under investigation are locust bean gum and starch.

C. Plans:

1. Produce machine made cigarettes with guar bound sheets and varying levels of DAP to evaluate cigarette performance (CI, subjectives).
2. Scale-up the "hybrid" sheet formulation and produce larger quantities of sheet in the Cast Leaf Lab for physical and subjective testings.

3. Continue the tobacco-binder interaction studies with TSA and U.S. tobacco feedstocks to produce sheets with improved physicals, burning and subjective characteristics.
4. Continue the screening of such other binders as locust bean gum, xanthan, starch and konjac flour for improved physicals and subjectives.
5. Determine the effects of cellulose fiber to sheet physical properties and subjective characteristics with factory dust feedstock.

II. ENCAPSULATED MENTHOL

- A. Objective: Provide technical assistance and formulations to Development Engineering for the microencapsulation of menthol.
- B. Results: Several stable emulsions of menthol in water were developed. One of these emulsions with 40 parts menthol in 90 parts water was selected for further testing by Development Engineering.
- C. Plans: Continue to provide assistance on an as needed basis.

PROJECT NUMBER : 1812
PROJECT TITLE : New Expanded Tobacco
PROJECT LEADER : E. B. Fischer
PERIOD COVERED : February, 1991

I. BATCH GASEOUS CO₂ IMPREGNATION

- A. Objective:** Define process parameters for a batch gaseous CO₂ impregnation process.
- B. Results:** Impregnation and expansion tests were conducted on the 8 inch tower to produce various expanded tobacco types (liquid CO₂ control, cased and uncased feedstock using gaseous impregnation, plus several casing schemes, again using gaseous impregnation) for cigarette making. Standard gaseous impregnation conditions of 15 percent OV tobacco, in-situ flow through cooling to 10°F, and 800 psi impregnation pressure were used. These conditions resulted in a tobacco post vent temperature of about -10°F.

The expansion results of each of the gaseous impregnated fillers compared favorably with the liquid CO₂ control runs. The SV of the expanded filler ranged from about 2.9 to 3.2 cc/g compared to about 3.0 for standard DIET. Stability of the impregnated filler appeared to be adequate. CV's above 9.5 were typically obtained for the uncased tobacco feed. The expansion was less for the cased tobacco feed. CV's of only 8 to 9 were obtained.

A successful preliminary trial was conducted at the Bermuda Hundred Facility on February 15 to check out modifications to accommodate gaseous CO₂ impregnation. Based on the success of the trial run, software modifications were made to improve the operation and control of the gaseous impregnation process. A series of four runs was conducted February 26-28 to obtain an assessment of the degree of impregnation as well as tobacco survivability through the system.

- C. Plans:** Evaluate the data from the Bermuda Hundred runs and conduct additional tests to further define operating system modification requirements.

II. CONTINUOUS IMPREGNATION PROCESS

- A. Objective:** Develop a continuous impregnation process to improve the subjectives of expanded tobacco while maintaining equivalent cigarette filling power to the existing process.
- B. Results:** Work has been initiated to conduct a comparative study of the CO₂ handling requirements for the three continuous impregnation concepts being developed: 1) high pressure rotary valve, 2) linear pocket processor, and 3) conveyor impregnation process. The results of the study will be utilized in the selection of a continuous impregnation concept for prototype development.

- C. **Plans:** Complete the comparative study of the CO₂ handling requirements for the continuous impregnation concepts.

III. ALTERNATE CONTINUOUS IMPREGNATION PROCESS

- A. **Objective:** Evaluate the linear pocket processor (LPP) for continuous impregnation of tobacco filler.
- B. **Results:** Tobacco degradation from feed to the cooler through the discharge from the LPP was studied. Filler was processed at 15 percent and 18 percent OV and at -10°F and 10°F for sieve analysis. Data and results are being analyzed.

Several areas of improvement for feeding the LPP were noted during experimentation. As throughput is increased from 200 to 600 lbs/hr, increased amounts of filler become trapped in the piston seals. Modifications are being made to clean the seals and alter the geometry of the feed section.

- C. **Plans:** The model LPP will be used to evaluate tobacco feed and discharge design parameters for the prototype LPP.

IV. TOBACCO COOLING

- A. **Objective:** Develop a continuous cooling process in support of the gaseous CO₂ impregnation program.
- B. **Results:** Process conditions for achieving -10°F and +10°F tobacco at a throughput of 500 lbs/hr were defined. Uniform exit temperature was obtained. The unit has been incorporated into the 8 inch pilot system for evaluation with the batch gaseous impregnation process.
- C. **Plans:** Widen the belt within the unit to reduce impregnator fill time. Continue test work with the cooler close coupled to the impregnator.

V. ALTERNATE PUFF/DRY/SET PROCESSES

- A. **Objective:** Define alternate means of puffing, drying, setting, and reordering impregnated tobacco to improve product subjectives and physical characteristics relative to the present DIET process.
- B. **Results:** Humid air conditions for reordering with minimum CV loss are being resolved in laboratory studies. Dew point condensation was shown to significantly contribute to collapse if not avoided by proper control of air conditions or tobacco temperature.
- C. **Plans:** Continue laboratory studies to resolve feasibility of air reordering. Follow Frigoscandia's development efforts and test their unit in late March. Evaluate the Cardwell air/water spray unit which is expected to be delivered by the end of March.

VI. TOWER HEAT TRANSFER RATES

- A. **Objective:** Develop process information to define heat transfer parameters for the design of a new expansion tower. Test process concepts on the 8-inch tower leading to process and product improvement.
- B. **Results:** The positive displacement feed valve design modifications have been completed. Plans are to install the modified valve assembly on the eight (8) inch tower. The cooling system will use liquid CO₂ at 100 psi to obtain subzero temperatures. Preliminary testing is scheduled for the week of March 4, 1991.

The mass spectrometer on-line monitor for tower gas analysis was operated for two weeks around-the-clock. Tower gas samples were taken at three different steam concentrations to complete the calibration.

A new tangential separator, modified to maintain the momentum of tobacco solids directed to the discharge rotary valve has been installed. The volume of the solids discharge section was significantly enlarged which should cut down on turbulence and help to obtain plug flow separation. Testing is scheduled for the week of March 4, 1991.

Heat transfer calculations using Dr. Zenz's correlation between Nusselt and Reynolds numbers were done for various tower lengths and at different tower conditions. Higher gas velocities result in higher extent of heat transfer, more than offsetting the reduction in residence time. Hence, for the same heat transfer, the tower length can be shorter when high gas velocity is used.

- C. **Plans:** Install and test the modified positive displacement feed valve and the new tangential separator.

VII. CHEMICAL STIFFENING

- A. **Objective:** Define a process to chemically stiffen expanded tobacco which will reduce thermal treatment and the associated subjective degradation while maintaining cigarette filling power equivalent to the current process.
- B. **Results:** A comparison between products stiffened with 2.7 percent calcium acetate consistently showed that those products made on the 3 inch tower at 400°F, 100 percent steam had a 15 percent lower acetic acid content than those produced in the 8 inch tower at 400°F, 75 percent steam. This may explain why the presence of an acetic odor was not noted during tests on the 3 inch system. In addition, lab scale tests sweeping air through the product suggest that the acetate (acetic acid) is held differently by gaseous impregnated tobacco and liquid impregnated tobacco. This is indicated by the fact that the acetic acid content was reduced with air sweeping through the liquid impregnated sample with 2.7 percent add-on of calcium acetate while no change in acetic acid content was shown with the gaseous impregnated sample even after air sweeping for 72 hours. Tests are planned to confirm this and evaluate the effect of all processing steps on acetic acid reduction.

A second pilot test to evaluate the effect of casing with a 2.7 percent add-on of calcium acetate as a stiffening additive showed that the presence of casing had no effect on the performance of the additive. CV improvements with the additive of about 1.5 cc/g were confirmed.

To investigate the role of sugars in the stiffening effect, two samples of burley tobacco were treated, one with 2.7 percent calcium acetate and the other with 5 percent calcium hydroxide and run through the expansion process. No stiffening effect was observed for either. However, with the application of 10 percent glucose to the burley before 2.7 percent calcium acetate, a stiffening effect of 1.0 cc/g CV improvement was seen when compared to a control with only 10 percent glucose added. This needs to be studied before conclusions are reached about the importance of sugar.

- C. Plans: Develop a profile of CV and add-on level for calcium acetate. After profiling the acetic acid removal ability of various processing steps, conduct tests with maximum potential for acetic acid reduction and evaluate subjectively to determine the acceptable threshold. Evaluate alternate systems which may have subjective advantages over calcium acetate. Conduct tests to evaluate the addition of a stiffening additive on strip prior to cutting. Complete tests to support mechanism studies.

PROJECT NUMBER : 0008
PROJECT TITLE : Computer Applications Division
WRITTEN BY : John Palesis and John Blankinship
PERIOD COVERED : February, 1991

I. Neurocomputing

- A. **Objective:** Develop programming techniques for use of the HNC ANZA Plus Neurocomputer and Neurosoftware. Investigate potential applications of neurocomputing to R&D problems, and apply where appropriate.
- B. **Results:** Research continued into several difficulties associated with backpropagation learning of multilayer networks. As manifested in the cigarette analyticals vs. liking problem and other mapping problems, these difficulties included: (1) extremely long training times with no guarantee of convergence to a good set of weights, (2) inability to train to a satisfactory mean squared error level, (3) overfitting of training data and poor generalization to new input patterns, (4) sensitivity to the initial set of weights and other learning parameters, resulting in significantly different predictions and standard errors depending on the random number seed, smoothing factors, etc., and (5) anomalous learning behavior.

The "MBPN Tool" control program and the training methodology for backpropagation learning were significantly enhanced to address these difficulties. The principal enhancements include: (1) testing mean squared performance during training with respect to a separate test data set to prevent overfitting of training data, (2) a cyclic mechanism for testing learning parameter adjustments during training, (3) allowing recovery after unsuccessful parameter adjustments and transient increases in the mean squared error, (4) a "stagnation monitor" to decrease the learning rate after the mean squared error has flattened out, (5) dynamically limiting the maximum learning rate, and (6) using an expanded training set. As a result of these enhancements, backpropagation learning is significantly improved in several respects. These include (1) faster convergence to a reliable set of weights resulting in lower mean squared error levels while still avoiding overfitting of training data, and (2) less sensitivity to the initial set of weights and other learning parameters.

Development of the Menthol Cigarette Liking Analysis Model (MCLAM), a neural network-based system for the analysis and prediction of liking ratings, was concluded. Using the enhanced backpropagation control program, a new neural network was determined for MCLAM. This network has five hidden layer processing elements, and exhibits a standard error of prediction of 0.43 on a seven-point liking scale. Also, a "Predict" option was added to MCLAM which determines predicted liking ratings against all smoker groups for existing or hypothetical menthol test products. The system is being used by PED in studies of menthol test products.

Using the HNC neurosoftware for Learning Vector Quantization (LVQ), a neural network technique for pattern recognition and classification was developed. LVQ iteratively determines a set of equiprobable representative patterns for each class

given a training set of examples. Combined with the use of the Parzen Window technique for computing a conditional class probability, LVQ exhibits near Bayesian performance. The technique was applied to the pattern classification of certain electrophysiological waveforms. Although nearly 100 percent classification accuracy was achieved with respect to the training patterns, classification accuracy was only around 70 percent for new waveform patterns. This was attributed in part to the considerable variability in waveform patterns within each class.

- C. Plans: Write a research report on the Menthol Cigarette Liking Analysis Model.

II. Expert Systems Development

- A. Objective: Develop an Expert System for Cigarette Design.

- B. Results: We are presently in the process of incorporating the new features of version 8.1 of the Fortran cigarette model into CigDES.1. This process essentially involves making changes and additions to (1) the interface between the Fortran mathematical model and the Lisp/KEE qualitative model and (2) the forward and goal-directed reasoning components of CigDES.1.

- C. Conclusions: The task of identifying the changes and additions from version 7.2 to version 8.1 of the Fortran model is greatly facilitated by the use of the UNIX System V Source Control System (SCCS). SCCS compares the two different versions and lists their differences.

- D. Plans: When this updating process is terminated, we will release CigDES.1.

- E. References: Palesis, J.A., Dwyer, R.W., Leister, D.L., and Kao, J.W., "Transforming Mathematical Product Evaluation Models Into Expert Systems for Product Design," Proceedings of the 3rd International Conference on Industrial & Engineering Applications of Artificial Intelligence and Expert Systems, pp. 404-415, 1990.

III. Machine Learning

- A. Objective: Apply AI-based machine learning in scientific research.

- B. Results: "Popcorn-nutty" Odor Study: As reported in the monthly report of November 1990, ID3, an example-based machine learning algorithm has been used successfully to identify the underlying chemical substructures of ACYL-PYRIDINES which cause the "popcorn-nutty" odor. In this study, the structures of the chemical compounds analyzed by ID3 were represented using a "positional" notation, that is, as a set of structural positions (the attributes) whose values were unit atoms. In view of the fact that converting chemical compound structures to this "positional" notation requires a great deal of effort on the part of the chemist, we wanted to explore the possibility of applying ID3 directly to a set of structures represented in the "smiles" notation. This notation is used by many chemical databases to store compounds

represented as strings of characters. The same set of compounds analyzed in the November study mentioned above were converted to the "smiles" notation and then processed by ID3. Unfortunately, the results were not satisfactory due to the fact that the ID3 algorithm analyzes the impact of single characters (i.e. columns in the relational table forming the "training set") on the result ("popcorn-nutty" odor in this case) and does not consider groups of characters (substrings) which represent meaningful units in the chemical compound structures.

- C. **Conclusions:** ID3 is not a proper algorithm for analyzing compounds represented with the "smiles" notation. A more promising way to discover the underlying patterns of compounds represented as strings is to apply pattern recognition techniques similar to those used in natural language processing.
- D. **Plans:** We will explore other methods for analyzing chemical compounds represented as strings to discover chemical substructures which cause specific chemical reaction.
- E. **References:** (1) J. Palesis. Artificial Intelligence Based Induction: A Case for the ID3 Learning Algorithm. Philip Morris R&D Technical Report, January, 1991.

PROJECT NUMBER : 1101
PROJECT TITLE : Entomological Research
PROJECT LEADER : D. L. Faustini
PERIOD COVERED : February, 1991

I. CIGARETTE BEETLE (CB) CONTROL PROGRAM

- A. **Objectives:** (1) To determine if phosphine resistance is occurring in the feral CB, (2) to determine if physiological differences are present in laboratory and field colonies regarding methoprene, and (3) investigate alternatives to conventional tobacco pesticides.
- B. **Results:** A bioassay has been initiated using F₂ larvae collected from Kenbridge and Kabat® feral cultures to determine if methoprene physiological differences exist (1). A second bioassay has been initiated to determine if Nylar® could show physiological differences in the local Kabat®-feral CBs. Nylar® is an insect growth regulator with a different chemical structure from Kabat® (2). Laboratory reared CBs are being used as controls in both studies.
- C. **Plans:** Complete bioassays (1) showing effects of methoprene concentrations on two types of feral CBs, and (2) determine the effects of Nylar® on CBs previously exposed to Kabat®.
- D. **References:**
1. Minor, M. F. Notebook No. 9024, pp. 15-17.
 2. Minor, M. F. Notebook No. 9024, pp. 22-23.

II. SERVICE TO OTHERS

- A. **Objective:** Provide technical services to areas outside R&D.
- B. **Results:** At the request of Engineering Department personnel, two tests were run to determine CB kill and temperature profiles using the Datatrace® temperature monitoring units in the oriental moisturizing cylinder (OMC) at the RLPPF. One hundred percent mortality was observed for all life stages of the insect (1).
- C. **Plans:** Issue memo detailing results.
- D. **Reference:**
- Lehman, R. M. Notebook No. 9014, pp. 34-39.

PROJECT NUMBER : 1720
PROJECT TITLE : Analytical Microscopy
PROJECT LEADER : V. L. Baliga
PERIOD COVERED : February, 1991

I. REDUCED SIDESTREAM/FILTRATION/PAPER TECHNOLOGY

(Thompson, Sanders, Miser, Baliga)

- A. **Objective:** Examine the ultrastructure of selected cigarette papers, paper additives, and filtration materials in support of the paper technology project.

B. **Results:**

Powders: Six samples of experimental filler for cigarette wrapper papers were examined for morphology and elemental content. Three of the samples were Reheis $\text{Mg}(\text{OH})_2$, all of which exhibited platelet morphology. However, the heated sample appeared to be slightly larger along one dimension while the third sample, a paste, appeared as ovoids instead of the hexagonal tablets of $\text{Mg}(\text{OH})_2$.¹ The fourth sample, made from $\text{Mg}''\text{Me}''\text{CO}_3$, consisted of particles similar in morphology to hydromagnesite ($\text{Mg}_5(\text{CO}_3)_4 \cdot 4\text{H}_2\text{O}$), and nesquehonite ($\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$) or lansfordite ($\text{MgCO}_3 \cdot 5\text{H}_2\text{O}$).² A fifth sample, made from hydromagnesite contained Mg-containing rhombohedra similar to magnesite (MgCO_3) and well defined hexagonal tablets similar to brucite ($\text{Mg}(\text{OH})_2$).³ The sixth sample consisted of Mg-containing rhombohedra with a Ca-containing

In response to competitors claims of whiter cigarette wrapper paper and improved freshness of cigarettes, Winston cigarette wrapper papers from October and November productions were examined as well as package overwrap films from December, 1989, and October and November, 1990. Cigarette wrapper papers from both the October and November production samples contained wood pulp fibers. The filler in the October production consisted of typical barrel-shaped calcite while the filler in the November production paper consisted of two phases of CaCO_3 , the rod-shaped aragonite and barrel-shaped calcite.^{5,6} This was confirmed by electron diffraction⁶ and later by x-ray diffraction.⁷ Interestingly, other November production Winston cigarettes contained only calcite CaCO_3 .^{7,8} Package overwraps from the October and November 1990 productions were similar to each other with a well defined crystalline orientation as indicated by high birefringence by polarized light microscopy. The overwrap from the December 1989 sample showed no orientation.⁵

C. **References:**

1. Sanders, K., "Examination of Three Reheis Samples," Memo to J. Fournier, February 11, 1991.
2. Thompson, L., and Baliga, V., "Examination of Mg-Containing Powder," Memo to K. Podraza, February 12, 1991.

3. Sanders, K., "Examination of Sample 8770-86B," Memo to J. Fournier, February 13, 1991.
4. Sanders, K., "Examination of Sample #9040-23," Memo to J. Paine, February 14, 1991.
5. Baliga, V., Miser, D., and Thompson, L., "Comparison of Winston Cigarette Wrapper Papers and Winston Package Overwrap," Memo to P. Grantham, January 28, 1991.
6. Miser, D., "Electron Diffraction Studies of Winston Cigarette Paper Fillers," Memo to P. Grantham, January 24, 1991.
7. Fournier, J., "Calcite and/or Aragonite: On the Consistency in the Characterizational Results," Memo to E. Sanders, February 11, 1991.
8. Baliga, V., PM Notebook 8911, p. 45, February 11, 1991.

II. SUPPORT TO R&D (Thompson, Sanders, Baliga)

A. Objective: To provide support for continuing programs within R&D.

B. Results:

Expanded tobaccos that had been sprayed with either $\text{Ca}(\text{OH})_2$ or Ca acetate, at 5%, were examined for morphological differences. The $\text{Ca}(\text{OH})_2$ sprayed tobacco was covered with $\text{Ca}(\text{OH})_2$ crystals on the outer surfaces while the Ca acetate sprayed tobacco had only a few crystal clusters that resembled acicular Ca acetate crystals. No evidence of Ca-containing crystals were found in the interior of the tobacco shreds. No obvious differences in cellular shape were noted among the two Ca sprayed samples and the control.¹

Other samples that were examined included the thickness of sputtered gold which was determined to be linear as a function of increasing sputtering time², metal slivers from the BL plant which were found to contain mostly Fe or Fe and Zn³, and fibers from Tunnel I insulation which were confirmed to be fiber glass.⁴

C. References:

1. Thompson, L., "Scanning Electron Microscope (SEM) Analysis of Control and Treated DIET Samples," Memo to J. Crump, January 28, 1991.
2. Sanders, K., "Thickness of Sputter Coated Gold," Memo to D. Kellogg, February 4, 1991.
3. Baliga, V., "Elemental Determination of Metal Pieces from BL Plant," Memo to D. Watson, January 30, 1991.

4. Baliga, V., "Tunnel I Insulation," Memo to M. Griff, February 11, 1991.

PROJECT NUMBER : 1752
PROJECT TITLE : Molecular Structure Determination and Materials Evaluation
PROJECT LEADER : G. Vilcins
PERIOD COVERED : February, 1991

I. INSTRUMENTAL ANALYSES OF SIDESEAM ADHESIVE

- A. **Objective:** To determine the proper instrumental procedure for the characterization of sideseam adhesives.
- B. **Results:** Four Fuller, four National, and one Ajax sideseam adhesives were analyzed by GC-FTIR, TC-MS, NMR, FTIR, MS, TGA, and X-Ray Fluorescence techniques. The results were evaluated to determine the procedures to be used for the characterization of the adhesive chemical composition.
- C. **Conclusions:** From these analyses it was determined that all four Fuller's adhesives, the AD-006 National, and the Ajax adhesives were formulated from polyvinyl acetate homopolymers. The other two National adhesives, AD-002 and AD-005, were formulated from polyethylene vinyl acetate copolymers. From this study it was concluded that for the adhesive chemical composition characterization only the NMR and FTIR instrumental analyses will be performed as they provided sufficient information on the adhesive chemical composition.
- D. **Plans:** No further work is planned at this time.
- E. **References:**
1. Bassfield, R., PM Notebook #7398, p. 179.
 2. Chung, C., PM Notebook #9035, pp. 24-28.
 3. Griff, M., PM Notebook #9042, pp. 19-20.
 4. Jensen, N., PM Notebook #8910, p. 85.
 5. Lyons-Hart, PM Notebook #7820, pp. 75-83.

II. METHOPRENE IN CARBON DIOXIDE

- A. **Objective:** To determine the ability of liquid carbon dioxide to remove methoprene from tobacco.
- B. **Results:** Experiments were performed in which methoprene-treated tobacco was subjected to temperature, pressure, and CO₂ conditions in a model process. Methoprene levels in ground tobacco samples were reduced from 3.6±0.2 ppm to 1.5±0.3 ppm when the tobacco was emersed in liquid CO₂ (28 deg. F, 600 psi) for 30 minutes. In order to see if any methoprene could be recovered and positively

identified, 15 mgs of methoprene were added to a Whatman #40 filter paper and placed in a simulated impregnation vessel. A sample of 25 ml of liquid CO_2 was pumped through the vessel and collected in 2-propanol. The 2-propanol was analyzed using supercritical fluid chromatography (SFC) with both flame ionization (FID) and infrared (FT-IR) detection. The FID on the SFC was used to quantify the amount of methoprene.

C. **Plans:** Based on the results from the laboratory model system, further experiments will collect liquid CO_2 from the DIET impregnator in the MC Primary.

D. **Reference:**

Thomas, E., Notebook #8492, pp. 65-66, 68-78.

III. MS ANALYSIS OF SF_6

A. **Objective:** To determine if SF_6 is present in trace amounts in competitors expanded tobacco.

B. **Results:** The requirements of the analysis for a highly specific method for a very volatile compound at trace levels from a complex mixture led to the development of methodology based on selected ion monitoring mass spectrometry. The study involved three major steps which included: 1) Determination of suitable ions and conditions for selected ion monitoring of SF_6 . 2) Exploration of possible interfering species from the tobacco matrix. 3) Analysis of sample and suitable controls.

C. **Conclusions:** The results show that SF_6 can be detected at ppb levels in expanded tobacco. However, no SF_6 was detected in the sample of interest.

D. **Reference:**

Jensen, N., "Mass Spectral Analysis of SF_6 ," Memo to D. Leyden, February 3, 1991.

PROJECT NUMBER : 1757
PROJECT TITLE : Analytical Flavor Specifications
PROJECT LEADER : M. L. Zimmermann
WRITTEN BY : B. Baronian/K. Dudzinski
PERIOD COVERED : February, 1991

FLAVOR SPECIFICATIONS AND CERTIFICATION

A. Objective: To develop analytical and sensory specifications for current, incoming flavor materials and to transmit specifications and methods for monitoring specifications to appropriate groups. To certify that PMI export flavor materials meet GFO, to issue a certification of analysis and to transfer methodology and certification to the Flavor Center. To develop specifications and analytical methods for adhesives used by PM USA.

B. Results:

Completed the specification process for the entire set of DM codes for all of the 92 flavor vendors for Philip Morris. Complete packages including methods specific for the items for each vendor are being prepared for distribution.

The certification program has been turned over in its entirety to the Flavor Center personnel. Responsibility for the certificate has also been moved to key individuals at the Flavor Center. Some problem solving and backup support continue to be addressed.

Efforts have been initiated to address issues associated with PMI samples for Malaysia. Several methods for the determination of key components have been completed and others are being investigated.

HPLC analysis for sugars on extractions of various dry flavors was performed to determine acceptable levels of roasting. Amino Acid determinations were conducted for specification development using an existing HPLC method. Support has been provided to BCR by analyzing for the nicotine content of two sets of tobacco samples.

Initial evaluations of sideseam adhesives from three different manufactures (Fuller, National, Ajax) and from three separate use locations (Manufacturing Center, Cabarrus, Louisville) are being investigated.

Contacts have been made with the technical services division of D. Sweeny through E. Stagg in which the flow from receiving/storage and use by each of the various makers and packers were examined.

A visit was made to the Richmond based H.B. Fuller Company in which the formulation of a generic PVAc/PVA adhesive was investigated.

Meetings have been held to discuss the proposed planned database for adhesives and the analytical results generated by the initial analytical investigations of sideseam adhesives.

C. Plans:

Continue support to the Flavor Center on an as needed basis, issue the completed Direct Materials specifications, and continue the visits with key vendors on technical issues.

Evaluate initial analytical results of sideseam adhesives. Continue to search for and develop applicable methodologies to determine the composition of adhesives.

PROJECT NUMBER : 1902
PROJECT TITLE : Tobacco Microbiology
PROJECT LEADER : D. M. Teng
WRITTEN BY : D. K. Chadick
PERIOD COVERED : February, 1991

I. TOBACCO MICROBIOLOGY

A. Objective: To develop methods and evaluate the microflora in tobacco materials.

B. Results:

1. 1990 Bright Tobacco Audit

To date, the microbial populations were enumerated from a total of 859 samples of bright tobacco (1,2). The data are being statistically analyzed.

2. Flavor Analyses

A total of four samples of a flavor were submitted for microbial analyses (Lot numbers 319184-07, 08, 09, 13). No microbial growth was detected in any of the samples (3).

3. Bactometer® Study

A completion report was issued (4). Since the instrument was determined to be an inadequate replacement for the plate-count procedure, appropriate disposal plans have been initiated.

4. Cut Filler Analyses - Special Request

A total of eight samples of cut filler were submitted for microbial evaluation (code numbers 1783, 4374, 4370, 2776, 5048, 4376, 3319, 624). The cut filler was initially export material; however, it had been stored in a warehouse since March, 1990. The bacterial and mold numbers were within the expected laboratory limits (5).

5. Microbial Quality Improvement Program (MQIP)

As part of the ongoing MQIP studies, samples were collected and microbially analyzed from a filter making run in the OC Make/Pack facility. The samples included aliquots from the filter tow, hot melt pellets, filter wrap, plasticizer, and wrap glue. No microbial growth was detected from any of the sampled items (6).

6. Effect of Moisture on Visual Mold Growth

A series of desiccators, with different saturated salt solutions to maintain a different relative humidity (RH) in each desiccator, were set up and kept in a walk-in chamber maintained at 37°C. Bright and burley ground strip from the 1989 crop (bright at about 10^4 mold colonies/g, and burley at about 10 mold colonies/g at zero time) were put into the desiccators. At 97% RH, the bright tobacco showed visible mold growth in 4 days, and the burley tobacco showed visible mold growth in 6 days. At 92% RH, bright tobacco showed visible mold in 7 days, and burley tobacco in 12 days. After 5 weeks none of the remaining tobacco samples incubated at 80, 70 and 58% RH showed visible mold growth (7).

C. **Plans:** (1) Continue statistical analyses and analyze an additional 90 samples of bright tobacco, analyze samples on an "as needed" basis, and continue the study on moisture vs. mold growth.

D. References:

1. Chadick, D. Notebook No. 9044, p. 9.
2. Chadick, D. Notebook No. 8904 pp. 165-174, and 178.
3. Chadick, D. Notebook No. 9044, p. 8.
4. Chadick, D. Completion Report 91-002; 1991 January 25.
5. Chadick, D. Notebook No. 9044, p. 9.
6. Chadick, D. Notebook No. 9044, p. 9.
7. Teng, D. Notebook No. 8788, p. 89.

II. NICOTINE DEGRADATION STUDY

- A. **Objective:** To develop methods for the biodegradation of nicotine.
- B. **Results:** Nicotine bioconversion was studied using different concentrations (MPC-nicotine was diluted with primary influent (PI) at 1:10 and 1:20) of feed and at different rates (1.25-day and 2.5-day retention times) in fermentors containing aeration basin (AB) material. The total suspended solids were maintained above the initial level by a sludge recycle. The nicotine levels in samples of the AB, PI, and MPC liquids along with mixtures of MPC plus PI at different ratios were analyzed at time zero. The amounts of nicotine were also monitored in the AB daily and in the secondary effluent (fermentor overflows) after 1, 5, and 10 days of activity. In addition, the bacterial populations from all the previously mentioned samples were enumerated at time zero and after 1, 2, 5, and 10 days of activity. Except Bu the

MPC-nicotine at a 1:10 dilution and a retention time of 1.25-days, the nicotine concentrations in the other fermentors were below preset concentrations (1).

C. Plans: Complete the analyses of TKN, BOD and off-gas.

D. Reference:

Tenhet, S. Notebook No. 8281, pp. 196-200.

PROJECT NUMBER : 1904
PROJECT TITLE : Tobacco Physiology and Biochemistry
PROJECT LEADER : D. J. Ayers
WRITTEN BY : V. S. Malik
PERIOD COVERED : February, 1991

LOW NICOTINE STUDY

- A. **Objective:** To investigate the biochemistry of the nicotine biosynthetic pathway at the putrescine N-methyltransferase (PMT) step and specifically to isolate PMT from tobacco root extracts.
- B. **Results:** Ammonium sulfate samples prepared from the roots of hydroponically grown burley 21 tobacco plants were fractionated on two 500 ml bed volume phenyl-Sepharose columns. PMT activities eluted off these columns were similar to the activities obtained from roots of plants from groups 25 and 26. The specific activity for the peak PMT samples was determined to be 88.9 units (1). Phenyl-Sepharose purified material was further processed by DEAE/AHS chromatography. Various low activity DEAE/AHS fractions from all previous harvests were pooled to obtain batch 5C (2).

Alkaline phosphatase (AP) was renatured according to the method developed by Delvaux et al. (3). However attempts to renature PMT have been unsuccessful to date (2).

Mouse anti-PMT serum was received from the contract lab (Pocono Lab, PA). This was the first bleeding since the animals were challenged with PMT. ELISA screening for the presence of the PMT antibody yielded positive results for sera from two mice immunized with putative PMT. Protein blots made from SDS-PAGE of batch 1 PMT were examined by antibody. The putative PMT protein band at 60 kD showed a strong positive reaction as visualized for the presence of primary antibody using an alkaline phosphatase secondary antibody conjugate (4).

The batch PMT material was resolved by SDS-PAGE and electroblotted. The PMT band was digested by cyanogen bromide (CNBr) (5,6) and sequenced by a vendor. The CNBr digest of the putative PMT band was resolved into a number of peaks by reverse phase HPLC (5).

Amplification of PR17 cDNA insert was attempted using T3 and T7 primers. A fragment of DNA of appropriate size was amplified (7). Cloning of the PR17 insert into vector pBI121 was also tried. The results are being evaluated (7). The degenerate oligonucleotide probes for putative PMT gene were labeled and hybridized to northern blots of poly A+ RNA prepared from tobacco roots. The results were inconclusive (8). Several cDNA clones (pVM1, pVM2, pVM3, PR7, PR12, PR19 and PR50) have been sequenced by using automated DNA sequencer and T3 dye-labeled primer (9).

- C. **Plans:** Harvest group 28 plants, and process the tobacco roots to the 40-65% ammonium sulfate stage. Continue to fractionate ammonium sulfate extracts by chromatographic methods. Continue efforts to renature PMT. Continue to sequence CNBr generated fragments of the putative PMT protein. Continue to sequence selected cDNA clones.

Attempt to amplify PMT-like sequences using degenerate primers.

D. References:

1. Lyle, J. Notebook No. 8856, p. 200.
2. Turner, D. Notebook No. 8973, p. 200.
3. Delvaux, A.; Lemos, M.; Moreau, C.; Erneaux, C. Regeneration of enzymatic activity after sodium dodecylsulfate/polyacrylamide gel electrophoresis and zinc acetate staining: The example of inositol 1,4,5-triphosphate 5-phosphatase. *Analytical Biochemistry* 188: 219-221 (1990).
4. Yu, T. Notebook No. 9002, p. 82.
5. Nakatani, H. Notebook No. 8384, p. 170.
6. Bower, P. Notebook No. 9032, pp. 16-17, 22-25.
7. Malik, V. Notebook No. 8974, p. 78.
8. Wahab, S. Notebook No. 8983, pp. 170-171.
9. Michalik, T. Notebook No. 9036, p. 77.

PROJECT NUMBER : 2500
PROJECT TITLE : Fundamental Chemistry
PROJECT LEADER : J. I. Seeman
PERIOD COVERED : February, 1991

I. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES
(Fournier, Howe, Kallianos, Paine, Podraza, Secor, Seeman)

- A. **Objective:** To develop inorganic materials for novel applications for reduced sidestream, for burn-rate modification, enhanced subjectives in cigarettes and for required properties in novel smoking materials.
- B. **Results and Plans:** Two polymorphs of calcium carbonate, calcite (rhombohedral) and aragonite (orthorhombic), were identified by x-ray diffraction as the filler in wood pulp papers from Wattens and a Winston cigarette. The Wattens paper contains approximately 73% aragonite and 27% calcite and the paper from this particular Winston cigarette contains approximately 56% aragonite and 44% calcite. X-ray diffraction of paper from another Winston cigarette showed the presence of only calcite. Full details of these results were reported in memo form. Further samples are expected for phase identification.

Five magnesite-related samples were prepared and submitted for handsheet making; 1) magnesite, prepared by hydrothermally treating hydromagnesite with potassium bicarbonate at 200°C for 48 hours, 2) magnesite, prepared by hydrothermally treating nesquehonite at 180°C for 48 hours, 3) 85% magnesite/15% $\text{Mg}(\text{OH})_2$, prepared by hydrothermally treating hydromagnesite at 180°C for 48 hours, 4) magnesite/eitelite, prepared by hydrothermally treating hydromagnesite with sodium bicarbonate at 180°C for 48 hours, and 5) magnesite/eitelite, prepared by hydrothermally treating eitelite at 180°C for 48 hours (the reaction was incomplete). Importantly, these preparations were designed to be achieved without CO_2 pressure.

Smoking results of cigarettes with papers containing 85% hydromagnesite/15% $\text{Mg}(\text{OH})_2$, prepared by hydrothermally treating hydromagnesite at 200°C for 48 hours, were received. A reduction of 61% with a static burn time of 9.2 minutes and a reasonable ash was obtained. These results are similar to those of cigarettes with papers containing magnesite samples produced using CO_2 over-pressures. Subjective evaluations are pending.

Subjective evaluations of three low sidestream sol-gel derived models have shown one model to be a promising candidate. This model was made with a filler mixture containing 15% [51% hydromagnesite/49% $\text{Mg}(\text{OH})_2$] and 15% CaCO_3 Multiflex MM. It showed a 68% visibility reduction and 9.4 min. SBT. Pending additional smoking and subjective evaluations, selection of the most promising candidates will be made for potential scale-up considerations.

Smoking results from a cigarette model, the paper of which was derived from the aqueous sol-gel route containing 11% [88% hydromagnesite/12% $\text{Mg}(\text{OH})_2$] and 19% CaCO_3 Multiflex MM, showed 78% reduction in sidestream visibility with a long

SBT. The long SBT was likely influenced by a rather high level of K_2 succinate flux, 9.75%. A request is being submitted for a remake of a similar handsheet with a lower level of fluxing agent for reevaluation of this filler mixture.

A composition containing 85% hydromagnesite and 15% magnesium hydroxide synthesized via magnesium methyl carbonate exhibited good sidestream smoke reduction (58%) and excellent subjective results while only containing a 6% level of the magnesium based inorganic filler. A large scale synthesis of one of the most promising magnesium methyl carbonate procedures was investigated. When this material was reacted with water and 1 equivalent of potassium hydroxide, on approximately a 1.5 liter scale, it generated the above mixture of hydromagnesite and magnesium hydroxide (85%/15%). When the identical reaction was conducted on a 4.5 liter scale only hydromagnesite was obtained. Based on this result and our previous experience with the "sol-gel" procedures, manipulation of experimental procedures will be required for scale-up.

A considerable part of our current efforts is being devoted to the special papermaking challenges presented by sol-gel derived fillers. In these pursuits we have consulted with Dr. David Kraske of the University of Maine. An empirical approach is planned to circumvent the gelling tendencies of the particles over the forming mat in the handsheet mold. In progress are studies to evaluate the effect of filler particle size and filler surface charge on filler retention during handsheet making. The particle size most likely to be important in this aspect is that of the aggregated or flocculated precipitate, as measured by the Malvern Particle Size Analyzer, rather than the unit particle size, which is normally measured by SEM, and is smaller than the former.

Particle size measurements have been made on samples derived from mag carbonate sol-gel preparations under different precipitation conditions. Preliminary indications are that flocs of particles precipitated from methanolic sols with either KOH or water are smaller than flocs precipitated, respectively, from methanolic gels. Precipitates from dilute sols form smaller flocs than those from more concentrated sols, regardless of the nature of the sol, i.e. methanolic or aqueous. Particles prepared by evaporation of the solvent from a gel tend to be relatively large and to consist predominantly of nesquehonite.

A new FT-IR technique has been developed which allows the qualitative analysis of magnesium hydroxide and hydromagnesite in cigarette papers. In this procedure approximately a 0.5 cm x 0.5 cm piece of paper is saturated with mineral oil, placed on a sodium chloride plate, and a FT-IR spectrum is acquired. The region between 3800 and 3550 cm^{-1} is expanded to more clearly show the hydroxyl stretching frequency for magnesium hydroxide ($\sim 3650 \text{ cm}^{-1}$) and hydromagnesite ($\sim 3700 \text{ cm}^{-1}$). The lower limit of detection of either component in a mixture is $\sim 10\%$. This method was used to confirm the presence of magnesium hydroxide in RJR's low sidestream cigarette paper.

Eitelite was synthesized under conditions involving four times more water than usual, relative to the magnesium throughput, and it was determined that particle size was not significantly affected.

The four "standard" proportions of calcium oxide to magnesium bicarbonate derived from hydromagnesite were repeated, this time with cooling to 10-15°C in an attempt to minimize aragonite formation. The resulting slurries were submitted for handsheet making, and sampled for characterization. This work will be extended to higher magnesium/calcium ratios by the use of magnesium chloride additions to the reactions.

Analytical results have been obtained on papers which contain a mixture of calcium carbonate and magnesium based inorganic fillers. A comparison of two samples indicate a greater visible sidestream smoke reduction on the sample which contained the synthesized calcium carbonate compared with the sample containing the commercial Multiflex calcium carbonate. This advantage may result because the synthesized calcium carbonate has a rhombohedral morphology while the Multiflex calcium carbonate has a spherical morphology.

Two 2 kg samples of hexapotassium phytate were prepared at 13.7% and 17.2% concentration. Glass was used throughout the preparations, to avoid organoleptic contamination by the plastic vessels previously used. Plastic vessels had been normal, since the salt solutions needed to be stored frozen to minimize bacterial growth.

II. MISCELLANEOUS

(Howe, Paine, Podraza, Secor)

Pyrolysis studies were completed to establish yields and product distribution, particularly from the vanillin/ethylvanillin mixed salt. This showed the presence of a true solid solution, in which methyl-migration was 3 or 4 times more likely than ethyl-migration. Work continues on the preparation of various nicotine metabolites for R. Carchman. The final step in the preparation of 5'-hydroxycotinine was completed successfully and the material delivered. Efforts continue to prepare 5'-hydroxynorcotinine. Suggestions for the preparation of this material and the last requested metabolite were documented in a memo to R. Carchman. An HPLC analysis procedure for nicotine in aqueous solution was found in the literature and provided to Jim Baggett to be sent to Electrosynthesis, Inc.

Several aspects of the chemical hygiene plan are currently being addressed. The first involves training individuals about safe laboratory procedures. A program has been developed by Linda Guinn (Chemical Hygiene Officer) which includes the major safety issues. Training at R&D, using this program, will be conducted by the Divisional Safety Representative, Linda Guinn or Ken Podraza. The second major aspect involves developing a safe laboratory environment. This will include issues such as storage of chemicals and the use of appropriate safety equipment. Plans to accomplish the second major issue are being developed.

PROJECT NUMBER : 2501
PROJECT TITLE : Smoke Chemistry
PROJECT LEADER : R. A. Comes
PERIOD COVERED : February, 1991

I. SIDESTREAM SMOKE CHAMBER

A. Objective: Operate an environmentally controlled chamber to measure selected components of sidestream smoke.

B. Results: Chamber runs using five cigarettes per run to investigate aerosol properties of IM13 controls and of three models from the magnesium carbonate mechanistic study have been completed. The results indicated that the low sidestream models X6D0BPQ (paper - 63g/m^2 , 35% CaCO_3 filler, 8% KH_2PO_4 , 3% malonic acid, 4 Coresta) and X6D0BPU (paper - 42g/m^2 , 30% hydromagnesite filler, 11% potassium succinate, 12 Coresta) had substantially smaller mean particle diameters, 0.118 and 0.122 μm , respectively, than the control X6D0BPP (paper - 25g/m^2 , 25-30% CaCO_3 filler, 0.6% citrate, 34 Coresta) and IM13 cigarettes with mean diameters of 0.139 and 0.141 μm , respectively. The data represents an average of five runs each for the BP series cigarettes and six runs for the IM13 models. An improved dilution device is being investigated for sampling into the LASX particle size distribution instrument and this instrument has recently been returned after repair and recalibration by the manufacturer.

A chamber study to compare the new single wrapped Super Slims prototype with the current double wrapped model is under way in the chamber. Normal analyses are being carried out in support of the planned introduction of the single wrapped model. Smokings have been completed on the prototype CORESTA apparatus to generate mainstream and sidestream TPM and puff count data on these two models.

A memo has issued describing the efforts to improve ammonia collection and analysis. This investigation involved a comparison of acid washed silica gel tubes with the bubblers used in the past. These commercially available tubes may offer advantages in ease of analysis as well as in reproducibility and sensitivity.

Preparations are underway to initiate a new study in conjunction with the BCR Division to assist them in their collection and quantitative analysis of compounds of interest.

Efforts in anticipation of the new analytical study in conjunction with sidestream subjective evaluations continue. Contacts with Engineering personnel have initiated design work for chamber modifications to permit subjective evaluations. An initial subjective study is underway with ARD personnel to determine if sidestream subjective differences do exist between cigarettes of considerably different composition in the wrapper.

It has been reported that 3-vinylpyridine might be a good marker for gas phase constituents of sidestream smoke. This material is not commercially available due to

its propensity to polymerize. Attempted synthesis of a release agent to provide 3-vinylpyridine as a control is underway by another division member.

Major maintenance involvement occurred during this period. A blade broke and flew off from the ceiling fan. A new fan was installed. Service checks were carried out on the overall Honeywell system, on the condensor units, on several of the on-line analyzers and on the boiler and Borgwaldt smoking machine.

- C. **Plans:** Chamber runs will continue as required. Plans continue to be formulated to utilize the chamber to conduct a large scale investigation to address the issues related to sidestream odor and irritation and to simultaneously collect analytical data while conducting subjective evaluations. Trapping and analysis studies will continue.

II. SIDESTREAM SMOKE

- A. **Objective:** Conduct studies on sidestream smoke including; development of methods for collection and analysis of sidestream semivolatiles and gas phase; visibility determinations; analysis of selected materials relating to sidestream odor and irritation; development of proprietary products.
- B. **Results:** The Analytical Controls-Hewlett Packard 5890 multidimensional capillary gas chromatograph has been installed and is operational. Initial problems have been remedied. An initial study to determine utility was carried out with a flavor extract derived from XAD resin extracts from the ART water column. Samples were investigated to determine the location and size of certain flavor compounds. This was carried out by chromatographing the mixture with the precolumn, turning the hydrogen flow off to use the detector as a sniff port, rechromatographing to profile the odors and heartcutting one of the most significant odors to the secondary analytical column. It was found that this odor was located on the secondary column in an area with peaks almost too small to detect by FID. This demonstrates the organoleptic importance of peaks which are exceptionally small when detected analytically. A section of the primary column chromatogram containing some 98% of the detected peaks was found to contain virtually no odor. A nine second cut from the primary column, containing one or two broad peaks, was shown to contain as many as 40-50 peaks when rechromatographed on the secondary column, thus demonstrating the separative power of multidimensional chromatography.

The O.I.-H.P. gc/ms has been installed and is operational. It has been used in the conventional injection mode for several projects.

- C. **Plans:** The O.I.-H.P. and the Analytical Controls - Hewlett Packard systems will continue to be utilized in the study to attempt to determine analytical parameters responsible for sidestream subjective differences.

III. MISCELLANEOUS

1. Neutron radiography - The measurement of condensibles behind the cigarette coal has begun using IM13 cigarettes. Some interesting preliminary observations have been made with the initial smokings. The first density data taken immediately after the lighting puff has previously been used as the control or "zero" line and all subsequent data has been compared to this. During the recent upgrade of the system, the density data from the unlit cigarette was used as the "zero" line. The first results using this new procedure showed that there is a large density loss throughout the cigarette when the cigarette is lit (with a match). Presumably, water is stripped from the cigarette by the hot gases from the match. The effect is much greater than the small density losses seen during dry puffing. A series of replicates is being run to determine the magnitude of this effect. An automatic lighter will be added to permit lighting of the cigarettes directly in the beam.
2. Infrared camera - Two members of the Division attended an operator's training course on the new IR camera. A memo has been written describing the course content. The camera has been set up to examine low sidestream cigarette models versus controls. System improvements have been made including a more secure mount for the camera and an improved cigarette viewing cell. A new version of the software for analyzing the IR camera images has been ordered as has a new temperature readout for the calibration blackbody.
3. Pyrolysis gc/ms analyses were conducted in support of the menthol release and ART programs and to supply information on potential new materials for use with our products.
4. Analysis of aldehyde samples in support of BCR efforts was completed. A memo was issued describing this work.
5. Familiarization with the Perkin Elmer-Nelson Turbochrom software continues. The system in the lab has been connected to selected gc's and is being used to monitor FID and SCD (sulfur) signals from sidestream smoke samples. Upgrades for additional systems will be arriving and these will be installed prior to hooking up additional instrumentation.

PROJECT NUMBER : 2520
PROJECT TITLE : Flavor Research
PROJECT LEADER : Y. Houminer
PERIOD COVERED : February, 1991

I. FLAVOR RELEASE TECHNOLOGY

A. **Objective:** To investigate the synthesis and pyrolysis of various flavor release systems for use in new or improved products.

B. **Results:** We continue to explore the grafting of the trans-2,3-cyclic carbonates of glucose derivatives to other carbohydrates using relatively simple molecules as model compounds. Methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate was prepared from methyl 4,6-O-benzylidene- α -D-glucopyranoside and triphosgene in 60% isolated yield. Reaction of methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate and n-butanol was carried out in chloroform and pyridine. In both solvents, the expected two isomeric carbonates, methyl 4,6-O-benzylidene-2-O-butoxycarbonyl- α -D-glucopyranoside and methyl 4,6-O-benzylidene-3-O-butoxycarbonyl- α -D-glucopyranoside were obtained in about equal amounts. However, the rate of reaction was much slower in pyridine than in chloroform.

Reaction of methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate and phenethyl β -D-glucopyranoside was also carried out in chloroform and pyridine. In both cases, a complicated reaction mixture was obtained. The reaction in chloroform was analyzed and two components were readily identified as phenethyl β -D-glucopyranoside and methyl 4,6-O-benzylidene- α -D-glucopyranoside, the decomposition product from methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate. Two other products were also isolated and identified, namely methyl 4,6-O-benzylidene-2-O-(phenethyl β -D-glucopyranoside 6-O-carbonyl)- α -D-glucopyranoside and methyl 4,6-O-benzylidene-3-O-(phenethyl β -D-glucopyranoside 6-O-carbonyl)- α -D-glucopyranoside in about equal amounts. Other products present in the reaction mixture have not yet been isolated. The reaction in pyridine is much slower and some decomposition of methyl 4,6-O-benzylidene- α -D-glucopyranoside 2,3-O-cyclic carbonate to methyl 4,6-O-benzylidene- α -D-glucopyranoside is also observed. Analysis of other components is in progress.

We have received an ultrasound generator and its associated accessories and reactors. The instrument has been checked out and preliminary investigation is being carried out.

We continue to explore the synthesis and pyrolysis of various menthol release agents. The magnesium and calcium salts of (+)-di-O-acetyl-L-tartaric acid mono menthyl ester have been synthesized and submitted for TGA. The mono menthyl ester of glutaric acid has been prepared and submitted for NMR. A large scale preparation of mono menthyl itaconate has been carried out to make 440 grams of product. The method of synthesis was improved to give an 85% yield vs. previous yields which averaged about 60%. A set of trials were run with G. Keritsis on the use of mono menthyl itaconate as an encapsulating agent for menthol. The results of these trials

were very promising. Menthol was encapsulated by cocrystallization. It was shown that these crystals can be hardened and stabilized by surface formation of the calcium salt of the itaconic acid. A series of experiments have been planned to develop this concept and provide materials for evaluation.

A new class of menthol release compounds derived from beta-hydroxy carbonyl compounds is being developed. A first example has been prepared and characterized (CR-2956). This is the menthyl carbonate of methyl 3-hydroxy-3-phenyl propanoate. Pyrolysis GC/MS at 300° shows clean decomposition to menthol and methyl cinnamate.

The preparation of a flavor-release agent for 3-vinylpyridine is being pursued. The latter is a marker for gas phase sidestream smoke. Reduction of 3-acetylpyridine gave the expected alcohol. Reaction of the alcohol with oxalyl chloride gave the oxalate ester. Preliminary results indicate thermal decomposition by a radical process giving 3-ethylpyridine and cyclobuta [b]pyridine instead of the expected 3-vinylpyridine. The analogous menthyl carbonate is in preparation.

II. FILTER MATERIALS AND PAPER TECHNOLOGY SUPPORT

- A. **Objective:** To find outside suppliers for large quantities of new filter materials and inorganic paper additives.
- B. **Results:** Hydrothermal production of magnesite at R.S.A. has been suspended. Recent results have indicated that Hasteloy B-2, a high nickel alloy, may be being corroded during the synthesis, resulting in unacceptably high levels of nickel in the final product. While it was thought that R.S.A.'s large pressure reactor was manufactured solely from 316L Stainless Steel, on careful checking, it was determined that the reactor had Hasteloy B-2 baffels welded to the walls. Until it has been conclusively shown that the process does not attack Hasteloy B-2, no further work will be scheduled at R.S.A.

Four major and two intermediate suppliers of bulk quantities of the inorganic compounds required for production of eitelite were contacted. Samples and the appropriate technical information on their materials was requested and has begun to arrive.

A sample of partially acetylated wood pulp and surface deacetylated two have been received from Courtaulds. The material look good and handsheets will be made in the coming weeks.

PROJECT NUMBER : 2526
PROJECT TITLE : Greenhouse Operations and Cooperative Tobacco Variety
Evaluation Programs
PROJECT LEADER : R. T. Bass
PERIOD COVERED : February, 1991

I. GREENHOUSE STUDIES AND RESEARCH ACTIVITIES

(R. Bass, G. Newell G. West and J. Kang)

- A. **Objective:** Maintain the R&D Greenhouses, conduct plant research studies, provide greenhouse-grown tobacco materials for support of other R&D programs, and provide requested assistance for special projects.
- B. **Results:** As requested by Project 6906, tobacco seed for several varieties were supplied for use in tissue culture research. The varieties were burley KY. 14, bright Speight G. 28, and Nicotiana rustica, brasilia R 120, and Nicotiana rustica, brasilia ACC 48.

At the request of Dr. S. Haut of Project 6908, two samples (1 carton each) of machine made burley cigarettes were provided. One sample was KY. 14 control from the 1984 Regional Farm Test. This sample was low in total secondary alkaloids. The second sample was N-88 from the 1989 Regional Farm Test and was high in total secondary alkaloids.

For Project 2501 we have provided two cigarette samples for chamber experimentation. One sample was KY. 14 control from the 1987 burley Regional Farm Test and the other sample was NC-95 from the 1987 bright Regional Farm Test.

A greenhouse tour and presentation was provided for a group of New York office personnel from the PM Corporate Planning Division and the Sales Staff.

As requested by A.Frisch, R.S.O., a small quantity of C-254 tobacco leaf tissue was provided for a radioactivity assay. This tobacco was from the first cold plant growth chamber run made in 1972.

An inventory was completed for our refrigerated storage supply of regular tobacco and cigarette samples in the two large lab refrigerators. The controls on the Foster refrigerator were repaired in late January.

The greenhouse benches have been set up for the production of Hydroponic Burley 21 tobacco, Group No. 28. The transplanting of 52 plants has been completed and all plants are showing good color and growth.

At the request of and in cooperation with Dr. Pat Bower of Miller Brewing Co., we have set up an experiment on bench No. 1 in the Greenhouse. In order to provide supplemental light, two lamps have been placed over bench No. 1. One is a metal halide lamp and the other is a high pressure sodium lamp. Rhizomes of 4 hop plant varieties, Fuggle, Mt. Hood, Cascade, and Galena have been planted in 10 inch

plastic pots containing our regular sand-vermiculite mixture. These have sprouted and are showing good growth. These plants will be grown large enough to provide adequate material for the planned research studies by Dr. Bower.

The regular Greenhouse plant production activities have been completed, including the preparation of 495 gallons of nutrient solution, the application of insecticide, the performance of all culture practices, and the seeding and transplanting of K-326, Speight G-28, KY. 14, Bu. 21, N. rustica, Oriental, N. glauca, and 4 varieties of hops (Fuggle, Mt. Hood, Cascade, and Galena).

The regular Greenhouse operational and maintenance tasks have been done on schedule including the repair of the receptacles on the north end wall and the wiring for the auxiliary lamps over benches No. 1 and 2.

C. **Plans:** Greenhouse operations and maintenance will be continued along with all requested support activities.

D. **References:**

1. G. Newell, N.B. No. 8762
2. G. West, N.B. No. 8559
3. R. Bass, N.B. No. 8999

II. COOPERATIVE TOBACCO VARIETY EVALUATION PROGRAM

(R. Bass, G. Newell and G. West)

A. **Objective:** To participate in the cooperative tobacco industry program in order to assure that the quality of all new tobacco varieties meets or exceeds all requirements as defined by minimum standards program.

B. **Results:** The processing and cigarette making for the 1990 Burley Regional Farm Test samples has been completed. This includes the combined control, and entries #4,5,6,7, and 8. Cigarettes for each sample were shipped to the smoke flavor panels of four other participating tobacco companies for subjective evaluation. Samples were also submitted to our PM smoke panel for evaluation. The C.V. and O.V. determinations were completed and the values are shown below. No notable differences were observed.

SAMPLE CODE	FILLING VALUE* cc/gram	EQUILIBRIUM MOISTURE CONTENT* %
CONTROL	4.51	12.29
NO. 4	4.58	12.38
NO. 5	4.14	12.55
NO. 6	3.64	12.45
NO. 7	3.94	12.16
NO. 8	4.12	12.36

* = The standard equilibration conditions of 75°F and 60% RH were used.

These values have been reported to the chairman of the Burley Tobacco Variety Evaluation Committee, Mr. J. R. Calvert of the University of Kentucky. A meeting of this committee is scheduled for Feb. 28 at Lexington, KY. Also on that day a meeting of the Standards Subcommittee is to be held.

At the request of Dr. S. Haut, arrangements were made with the Leaf Department Agr. Programs for the field production of about 500 pounds of LAB-21 tobacco. This tobacco was grown at the Greeneville, TN. Tobacco Experiment Station using standard production practices. After harvest and curing, it was stemmed and a quantity of 800 pounds of blended strip representing all stalk positions was delivered to Dr. Haut for project 6908 studies.

- C. Plans: To continue to participate in the Cooperative Tobacco Variety Evaluation Program.

III. OTHER ACTIVITIES

1. Prepared report on Accomplishments for 1990.
2. Prepared and presented a Project Review for 1990.
3. Attended the Mold Symposium Meeting.
4. Attended R&D Strategy Plan Meeting.
5. Provided a copy of a 1966 report on Puffed Stem Slicing to P. Aument as requested.
6. Attended a meeting on the 1990 Burley Crop Satellite Survey given by the EOSAT Company in collaboration with PM Leaf Dept.

7. References:

1. R. Bass, N.B. No. 8999
2. G. Newell, N.B. No. 8762
3. G. West, N.B. No. 8559

PROJECT NUMBER : 2705
PROJECT TITLE : Tomorrow
PROJECT LEADER : R. W. Dwyer
PERIOD COVERED : February, 1991

I. PROJECT TOMORROW

- A. **Objective:** Explore the feasibility of developing a test for measuring the ignition propensities of cigarettes.
- B. **Status:** A test apparatus has been constructed in which the flow rate of air through a fabric/foam rig can be programmed. This apparatus was used with a variety of smouldering cigarettes to ascertain the flow rate necessary to cause smouldering ignition in a rayon fabric. Preliminary results suggest this method may give more reproducible results than previous flow methods, but many more tests need to be performed in order to obtain statistically significant results. We have also treated the rayon fabrics with a variety of burn promoters. The goal of this aspect of the work is to determine if the flammabilities of the fabrics can be adjusted so that we can discriminate between the ignition propensities of cigarettes.
- C. **Plans:** We plan to continue testing this apparatus using cigarettes designed to have mass burn rates ranging from 35 to 60 mg/min.

II. PROJECT TOMORROW

- A. **Objective:** Determine the thermal properties of cigarettes and substrates in order to correlate their design properties with their flammabilities.
- B. **Status:** A calorimetric method and apparatus have been constructed for measuring the heat outputs of cigarettes. Preliminary results show good correlations between cigarette heat flux and their mass burn rates. A method has also been developed to measure the effects of burn promoters on the ignition temperatures of rayon fabrics.
- C. **Plans:** A series of cigarettes has been obtained in which the properties of the wrapper have been varied systematically. These properties include permeability, chalk level, basis weight, and burn additive type and level. The heat outputs of these samples will be measured.

III. PROJECT TOMORROW

- A. **Objective:** Develop catalysts to reduce the delivery of CO of cigarettes.
- B. **Status:** A $\text{Pt/Fe}_2\text{O}_3$ catalyst was prepared according to instructions provided by Dr. Augustine of Seton Hall. The material was dried and calcined at 400° for 5 hours. The preparation was easy to implement and the catalyst showed good activity in oxidizing CO at room temperature.

- C. **Plans:** This catalyst is a fine powder. We are examining means of supporting it on larger particle-size materials in order to incorporate it into cigarette filters.

PROJECT NUMBER : 2706
PROJECT TITLE : New Expanded Tobacco
PROJECT LEADER : T. M. Howell
PERIOD COVERED : February, 1991

I. EXPANDED TOBACCO

- A. **Objective:** Determine if the CV of expanded filler can be improved and/or if post expansion collapse can be reduced by pretreating DIET feed with approved additives. Determine if these same additives improve the CV of Blend components.
- B. **Results:** The CV of Burley filler was increased by the addition of either calcium acetate or calcium hydroxide and the increase was to some extent greater than that observed for bright filler. A 1.3 cc/gm CV increase was attained on burley filler by adding 5% calcium hydroxide at 30% OV and bulking for 24 hours. A 0.5 cc/gm CV increase resulted with the addition of 3% calcium acetate. Applying 10% glucose to the burley prior to adding 3% calcium acetate did not yield any further improvement.

Results for the expansion of burley containing calcium acetate or calcium hydroxide were not consistent with those observed for the unexpanded filler. There was no improvement in CV in either case. The addition of 10% glucose to burley increased the equilibrated OV of the expanded filler yielding a CV of 10.2 cc/g. Combining calcium acetate with the glucose increased the CV to 11.1 cc/g at the higher equilibrated OV.

Applications of 0.5% calcium acetate and 0.4% calcium hydroxide to bright tobacco did not affect the stiffening of the filler.

- C. **Conclusions:** The addition of calcium acetate or calcium hydroxide to burley filler yielded similar improvements in stiffness as seen for bright. The addition of glucose did not alter the results indicating that the mechanism for stiffening is unlikely to be by the reaction of calcium with the sugars present. The presence of glucose did appear to make a difference when the burley was expanded. Further testing is necessary in order to clarify this.
- D. **Plans:** An analysis of all data collected to date is currently being made. Additional experiments are planned for optimizing stiffening conditions while minimizing additive amount, understanding the mechanism(s) for stiffening, and for evaluating other additive systems which may provide better subjectives.

II. EXPANDED TOBACCO

- A. **Objective:** Evaluate humid air reordering as a means for reducing post expansion collapse during reordering.

- B. **Results:** Experiments have been initiated to determine rates of reordering and extent of collapse in CV and SV as a function of temperature, humidity, air velocity and bed depth.
- C. **Plans:** Finish the initial experimental grid, analyze the data, and then recommend course for further testing.

III. EXPANDED TOBACCO

- A. **Objective:** Determine the minimum temperature at which stiffening of expanded tobacco occurs and at what temperature the tobacco must be subjected to afterwards in order to prevent collapse upon cooling.
- B. **Results:** DIET filler at 16% OVA which had been impregnated with liquid CO₂ in D Pilot Plant was used as stock to investigate the best thermal regimes for expansion and setting. Unequilibrated SV results showed significant improvement for samples exposed to thermal treatment on expansion when compared to a control expanded at ambient conditions. Expansion temperatures of 231° C for 7.0 seconds and setting temperatures of 134°C for 120 seconds have produced the highest SV measurements in these tests.
- C. **Conclusions:** Attempts at decoupling expansion from setting have been unsuccessful to date with the available laboratory techniques. As past experiences have indicated, the degree of expansion is directly related to rate and amount of heat input during expansion. Setting temperature is also difficult to establish due to the influence of tobacco moisture on the plasticity of the tobacco shred. Efforts to determine the minimum thermal environment required for setting will have to factor in the effects of expansion rate and moisture content while using the current laboratory testing procedures.

PROJECT NUMBER : 2707
PROJECT TITLE : Vision Inspection Technologies
PROJECT LEADER : R. J. Maher
PERIOD COVERED : February, 1991

I. PACK INSPECTION SYSTEM

- A. **Objective:** Develop and implement an on-line cigarette pack inspection system with the capability for global inspection.
- B. **Results:** Tuning and testing of the OSIRIS prototype which was installed at Stockton Street in mid-January has continued in preparation for a formal evaluation of the system's performance. The system has demonstrated its unique capability for detecting random defects such as crushed packs and printing errors. Adjustments to the vacuum conveyor to minimize pack wobble during image acquisition have reduced the nuisance reject rate to acceptable levels. A QA audit of rejected packs was carried out over a two-week period to estimate the nuisance reject rate. The rate was found to be 40% of the total reject rate during the first week and only 20% the second week. Two hardware problems remain to be resolved. The first is attributed to an error in the Matrox microcode which results in periodic system hangs. A corrected release of the Matrox software may eliminate this problem. The second hardware problem effects the performance of one of the three cameras. Efforts to address this problem are continuing.

The OSIRIS technology has been transferred to Itran in order to permit the development of a commercial Itran/OSIRIS inspection system based on Itran's new FS-11 image processing board.

- C. **Plans:** Continue efforts to eliminate hardware malfunctions. Install and test the low variance enhancement to the inspection algorithm. Initiate the formal evaluation of the system's performance.

II. PRINT WEB INSPECTION

- A. **Objective:** Develop a system for the global inspection of print web on the printing press.
- B. **Results:** The Guay/Dalsa camera data rate was increased to 20 MHz and a synchronizer circuit added to the camera/Trapix interface. The laboratory prototype web inspection system can now capture high resolution images from a surface moving at 400 ft/min. Substitution of a 512 element camera for our current 1024 element camera will permit operating at the target surface speed of 800 ft/min. A previously reported line interference pattern has been eliminated and the image contrast improved. An effort to quantify the variation in the image gray scale values has been initiated.

- C. **Plans:** Increase the surface speed capability to 800 ft/min. Complete the experiments on the gray scale variation. Test and refine the proposed inspection algorithms.

III. OFF-LINE INSPECTION

- A. **Objective:** Develop a system for the off-line inspection of print cylinder verification prints.
- B. **Results:** A detailed plan for the development of an inspection system has been prepared. The plan includes recommendations for both hardware and inspection algorithms along with a development schedule and cost estimates. The plan was presented to a group of personnel from Engineering Applied Technology, Development Engineering and the Vision Technologies Project. The plan was modified in accordance with several recommendations from the group.

An algorithm to align images relative to a master was developed and implemented using the Matrox 1280 imaging system.

- C. **Plans:** Review the plan with personnel from Colonial Heights Packaging. Prepare and submit appropriation requests.

PROJECT NUMBER : 6502
PROJECT TITLE : Process Monitoring and Real Time Measurements
PROJECT LEADER : K. B. Koller
PERIOD COVERED : February, 1991

I. GAS PHASE SMOKE ANALYSIS

- A. Objective:** Continue to develop quantitative multicomponent MS gas-phase smoke analysis by FT-IR spectroscopy.

B. Results:

Automatic data analysis and tabulation software has been written for a quantitative 12-component gas-phase MS smoke analysis procedure based on FT-IR spectroscopy. IM13 cigarettes were analyzed to establish a reference point for the evaluation of other cigarettes.

A study of the effect of aging on the concentrations of some of the volatile gases present in MS smoke was conducted. Hydrogen cyanide, methanol, isoprene, and acetaldehyde rapidly decreased in concentration with time. Acetylene, carbon monoxide, carbon dioxide, ethane, methane, ethylene, nitric oxide, and propylene concentrations remained constant or decreased slightly.

Individual calibration spectra of each of the above 12 gases were subtracted from a MS smoke spectrum. Analysis of the residual smoke spectrum revealed the presence of carbonyl sulfide and acetone. No other MS gas-phase components could be identified. This is due to either low concentrations, low IR activity, or the signal-to-noise constraints of collecting puff-by-puff spectra during a one minute smoking cycle.

- C. Plans:** Order and install the equipment necessary to provide for simultaneous analysis of MS and SS gas-phase smoke.

II. MS SMOKE STUDIES

- A. Objective:** Determine the effect of sizing agents in paper on MS smoke composition of cigarettes

- B. Results:** MS smoke condensates from the two cigarette models with different sizing agents on paper were analyzed chromatographically and compared. The control and test cigarettes were made with low sidestream paper, E2560. The control was sized with 6.6-7.4% potassium succinate and the test with 6.5-6.6% hexapotassium phytate. The condensates were fractionated into basic and neutral fractions. The acidic fraction was obtained by direct reaction of a portion of the condensate with a derivatization reagent (BSTFA) prior to the analysis. Close inspection of the data did not reveal any significant difference between the control and the test samples in their semi-volatile compositions. The acidic fractions from both the control and the test showed a significant reduction in the levels of formic acid, acetic acid and 3-

hydroxypyridine when compared to Marlboro Lights.

- C. Plans: To provide analysis of semi-volatile compounds present in smoke as needed.

III. R&D AND OPERATIONS SUPPORT

- A. Objective: To provide analytical support to R&D and Operations personnel and projects.

- B. Results:

The amount of methane and carbon monoxide present in stack gas emission samples were determined for J. Lephardt.

Development was initiated of a procedure to determine the level of nicotine present in an aerosol for Physical Research.

Development was initiated of a QA procedure for analysis of adhesive shipments to the Cabarrus facility.

- C. Plans: Continue to support R&D and Operations as need.

PROJECT NUMBER : 6503
PROJECT TITLE : Chemical Analysis
PROJECT LEADER : B. M. Handy
PERIOD COVERED : February, 1991

I. PAPER TECHNOLOGY

- A. **Objective:** To determine the elemental composition of compounds for use in experimental cigarette wrapper and on papers containing these compounds. Monitor the elemental composition of cigarette papers from the marketplace.
- B. **Results:** Papers containing various minerals such as eitelite, magnesite and calcite were analyzed using acid digestion with solution analysis by titration, inductively coupled plasma and atomic absorption. Choice of analytical technique is a function of constituent concentration. Papers from the CI were first qualitatively screened using XRF; when detected, calcium, magnesium, sodium and potassium were quantitated. Calcium, sodium, potassium and organic acids were determined on BOLD configuration papers to support studies on chalk type and additive levels on smoke results. Additional analyses were performed to support studies to determine the effects of variation in the sodium/potassium citrate ratios.
- C. **Plans:** Continue support as needed. Formalize analytical procedures for specific fillers and coatings with a high probability of continued use in experimental models or product.

II. CAST LEAF

- A. **Objective:** Determine alkaloids in burley stems and effluent from the BL Plant to support environmental studies. Determine alkaloids, phosphorus and ammonia in process liquors and cast leaf from the BL Plant to support material accountability studies.
- B. **Results:** Analyses performed were restricted to those tests not performed in QA. Procedures used routinely for Cast Leaf and tobacco were found to be applicable to materials from the various sampling points for this study. Current testing is complete.
- C. **Plans:** Support engineering studies as needed.

III. METHOPRENE (KABAT)

- A. **Objective:** Support engineering efforts of improve the uniformity of Kabat application.
- B. **Results:** Samples taken at spray nozzles from the center and sides of the spray application boom were analyzed to determine the spray composition at application start-up and during a run.

- C. Plans: Continue support as needed.

IV. CIGARETTE PAPER ANALYSIS

- A. Objective: Examine a competitors test market cigarette to determine the chemical components in the paper.
- B. Results: By comparison with analytical results obtained from papers of known composition removed from cigarettes, it was confirmed that this test product contained levels of acetic and malic acid suggesting additive use. There were no unusual findings based on the filler analysis; chlorides, nitrate nitrogen, PG/GLY and soluble ammonia, alkaloids and sugars were found at characteristic levels.
- C. Plans: Blend composition analysis is being performed.

PROJECT NUMBER : 6505
PROJECT TITLE : Special Investigations
PROJECT LEADER : D. F. Ingraham
PERIOD COVERED : February, 1991

R&D OPERATIONS SUPPORT

A. **Objective:** To provide analytical support to R&D and Operations personnel and projects.

B. **Results:**

Analyses of various packaging samples were provided for Purchasing Technical Services. Various additional solvents were quantitatively determined in support of establishing subjective thresholds by Flavor Development.

Various treatments for lowering the moisture content of cigarettes were investigated as part of a study to relate stale subjectives with chemical changes in low OV cigarettes. These experiments will be used to determine the specific conditions and times necessary to reach a target moisture level of 9%. The different treatments being investigated are heat, dessicant, sweeping with an inert gas, and vacuum.

PROJECT NUMBER : 6902
PROJECT TITLE : Biochemical Special Investigations
PROJECT LEADER : B. D. Davies
PERIOD COVERED : February, 1991

I. NICOTINE SPECIFIC MONOCLONAL ANTIBODY

- A. **Objective:** Develop a nicotine monoclonal antibody based enzyme-linked immunosorbent assay (ELISA) for the determination of nicotine concentrations in samples of interest to PM.
- B. **Results:** The Bio-Tek microtiter plate reader and microtiter plate washer have been received, installed and calibrated. Two experiments were conducted to assure that the plate reader is functioning correctly.

Using the ELISA analysis software supplied with the plate reader, two protocols were written to facilitate the complete analysis of ELISA data.

Several experiments were conducted this month to examine the amount of variation contributed to ELISA analyses by our tobacco extraction procedure. HPLC analyses performed by Project 6912, of replicate extracts of 250 mg of tobacco in 50ml of 1% triethanolamine (TEA) and our standard 500 mg of tobacco in 50 ml of 1% TEA were conducted and compared. The analyses indicated the replicate 250 mg extracts had a lower %RSD than the 500 mg extracts. The use of 250 mg tobacco extracts, with an appropriate change in the standard dilution prior to ELISA analysis, has been incorporated into the assay.

Using this extraction modification on tobacco from a new bottle of Grizzel Bright Monitor supplied by ARD, 18 replicate extractions were prepared and 10 of these were examined using both Nicotine ELISA and HPLC analyses (Project 6912). The average % nicotine value (mg/mg basis) and its associated %RSD for the ELISA data was 2.21% and 8.1%, respectively. For the HPLC data they were 2.20% and 4.29%, respectively. Both of these values were lower than the ARD (E-86b method) value of 2.5% determined for this same sample.

A preliminary experiment was conducted to examine the time course of nicotine extraction using our procedure. The results of that experiment will be reported next month.

- C. **Plans:** Complete the time course experiments. Determine the source of the difference between ELISA and ARD % nicotine values.

D. **References:**

1. Dunn, R. L. Notebook No. 9049, pp. 14-16.
2. Davies, B. D. Notebook No. 8638, p. 193.

3. Crockett, E. A. Notebook No. 8998, pp. 191-200.
4. Crockett, E. A. Notebook No. 9061, pp. 1-24.

PROJECT NUMBER : 6906
PROJECT TITLE : Biological Effects of Smoke
PROJECT LEADER : G. J. Patskan
PERIOD COVERED : February, 1991

I. JB6 MOUSE EPIDERMAL CELL TRANSFORMATION ASSAY (G. Nixon)

- A. Objective:** To obtain a colony formation response to 2R1 whole smoke treatment on 2R1 gas phase in soft agar.
- B. Results:** The soft agar assay testing the effects of 0.5 to 2 puffs of 2R1 whole smoke was completed. In this experiment, there was some toxicity at the 2-puff level, an effect not seen to the same extent in an earlier experiment which involved agar exposed to 4 puffs and subsequently diluted down to the 2-puff level. The original 4-puff experiment has been repeated. Two experiments testing the effects of 1-4 puffs of 2R1 gas phase (Cambridge filter assembly inserted into the smoking machine flow) were begun. Results of the first experiment did not show colony formation above control levels.
- C. Plans:** Obtain colony count data for the second 4-puff whole smoke and gas phase experiments.
- D. References:**
- Burruss, T. J. Notebook No. 8896, p. 36.
- Nixon, G. M. Notebook No. 8711, pp. 167-168.
- Vaughan, B. G. Notebook No. 8948, p. 111.

II. SALMONELLA/MICROSOME (S/M) ASSAY (D. Stagg)

- A. Objective:** To test the biological activity of experimental CSCs and other pertinent materials.
- B. Results:** Five CSC's were tested this month in support of the Cross Soluble Base Web studies. The results have been reported in memos.
- C. Plans:** Complete ongoing studies and continue to test samples for biological activity as they become available. Continue to evaluate the "screening" assay protocol as it is currently performed.

D. References:

Jones, R. Notebook No. 8769, p. 108.

Stagg, D. Notebook No. 9038, p. 114.

III. PLANT TISSUE CULTURE (M. Shulleeta)

A. **Objective:** To develop procedures for the establishment, maintenance and transformation of plant cell cultures.

B. **Results:** Procedures for the isolation and purification of *N. Tabacum* cv Burley 21 protoplasts have been established. In order to stabilize the protoplast membranes during electroporation and to determine optimal conditions for electroporation, experiments have been conducted to assess the effect of bovine serum albumin (BSA) addition to the medium at varying levels and varying voltage/capacitance settings. Preliminary observations indicate that the number of protoplasts which survive electroporation is higher in BSA treated preparations than in nontreated controls, however, quantitative results are not yet available to confirm these observations or to determine the voltage/capacitance conditions which result in at least 30% survivability.

Axenic cultures of *N. tabacum* cv Kentucky 14 and *N. tabacum* cv Speight G-28 have been initiated as additions to the stock cultures already maintained. Seeds for 2 *N. rustica* cultivars have also been obtained (Brazilia 48 and Brazilia R-120) for axenic culture generation.

C. **Plans:** Optimize the medium and voltage/capacitance conditions for electroporation of Burley 21 protoplasts.

D. References:

Shulleeta, M. Notebook No. 8961.

PROJECT NUMBER : 6908
PROJECT TITLE : Smoke Condensate Studies
PROJECT LEADER : A. H. Warfield
WRITTEN BY : W. R. Morgan
PERIOD COVERED : February, 1991

I. REDUCTION OF MS TSNA BY INHIBITING THE PYROSYNTHESIZED TSNA

- A. **Objective:** Reduce the levels of pyrosynthesized MS TSNA by blocking reaction pathways which form TSNA.
- B. **Results:** Oriental (Or) fraction #1 (insolubles) and n-docosyl stearate (DcS) were applied to SCF-Bu filler (JOBFO) in an attempt to reduce the MS delivery of TSNA. Or fraction #1 consists primarily of saturated high molecular weight hydrocarbons and DcS is a high molecular weight waxy ester. Both treated fillers showed reductions in TSNA relative to the control SCF-Bu but Or fraction #1 was much more effective. Reductions with Or fraction #1 were 42%, 73%, and 39% for NNN, NAT, and NNK respectively while DcS gave reductions of 24%, 39%, and 28%. Since SCF-Bu was used as the base substrate and the preformed TSNA have been removed in the SCFE treatment, the observed reductions are attributed to inhibition of TSNA pyrosynthetic mechanisms.
- C. **Plans:** Additional smoking experiments with other Or fractions are in progress.
- D. **References:**

Haut, S. A. Notebook No. 8891, p. 170.

II. ANALYTICAL PROCEDURES

- A. **Objective:** To develop and maintain analytical methodology for TSNA or other compound classes where information is needed for determining relationships of TSNA to their precursors.
- B. **Results:** Work continued on the development of a satisfactory smoke collection procedure for aged SS smoke. As was reported last month, the NaOH backwash step used in the normal nitrosamine workup procedure removed significant amounts of NA's when small amounts of smoke are present in the collected sample. This problem prevented testing of the new C-frit traps found to be satisfactory for collection of unaged SS smoke on the SAVA smoking machine. Tests were conducted this month using a modified sample workup procedure (elimination of the NaOH step) and the results indicated that this procedure rectified the nitrosamine loss problem. Tests were also conducted using a modified buffer (MB) solution in the collection traps to test for possible artifactual nitrosamine formation in the traps. The MB solution had an increased buffer capacity to prevent pH changes during collection and an increased ascorbic acid content. Both MB and standard buffer (SB) solutions were tested with and without added amine spikes (piperidine and

anatabine). The results of these tests showed that the new C-frit traps are satisfactory for collection of aged smoke and that artifactual nitrosamine formation in the traps is not a problem and that use of the standard buffer is acceptable.

Method development for pseudoxynicotine and N-methylmyosmine (PsON/NMM) in filler using the GC/MSD continued. Calibration was expanded from a single point calibration to a multi-level calibration. Experiments to determine the effects of solvent, mesh size of ground filler, filler to solvent ratio, and extraction time were outlined. The majority of time this month was concentrated on the extraction solvent. Preliminary results indicate that basic methanol is the best solvent. Four bases at four different concentrations are under investigation to ascertain the best base and concentration to be used.

- C. Plans: Extend the study of collection methods to solid phase traps. Continue development of the PsON/NMM method.

D. References:

Morgan, W. R. Notebook No. 8905, pp. 28-29.

Keene, C. K. Notebook No. 9067, p. 30.

III. UNEXTRACTED NICOTINE STUDIES

- A. Objective: To determine whether there is a correlation between unextracted nicotine in filler and the delivery of NNK in the corresponding MS smoke.

- B. Results: Work continued on optimizing the digestion procedure for liberation of Nic-X from burley CEL. The mild digestion procedure (1N NaOH at 40°C for 18 hours) reported last month has been adopted as the method of choice for all future digestions of CEL. Procedures for digestion of ART burley filler were begun.

- C. Plans: Continue evaluation of the ART burley filler method.

D. References:

Lambert, E. A. Notebook No. 8852, p. 191.

PROJECT NUMBER : 6912
PROJECT TITLE : Tobacco/Smoke Relationships
PROJECT LEADER : R. R. Izac
PERIOD COVERED : February, 1991

I. CROSS SOLUBLES BASE WEB (CHEMISTRY)

- A. **Objective:** To modify tobacco solubles and evaluate the smoke chemistry of fillers from various treated solubles and the appropriate base web.

- B. **Results:** In a study to evaluate different starting materials, burley CEL and burley S1 (BuS1) were treated with an ion exchange resin (AG50w-X). Model cigarettes from the batch treatments would not burn and samples of the filler were ground and submitted to ARD.

The soaking versus spraying study was repeated. The treated fillers were made into cigarettes and smoked. Cigarette smoke data showed that there were no differences in CSC the yields. Sample fillers were submitted to ARD and CSCs were submitted for S/M testing.

Testing continued with the new electrodialysis unit. The dialysis cell was repacked and modifications to the unit were made. Three tests to denitrate BuS1 have been conducted using a 10 (twice) and 15 DC volt setting. Nitrate test strips indicated that denitrification occurred at about 15 minutes at 15 DC volts.

BuS1 and BuI1 were separately treated with cellulase at 40°C for 19 hours. The samples were centrifuged and the supernatant is being freeze dried in preparation for application to base web.

- C. **Plans:** Continue to generate model fillers from various CSBW studies. Submit IT CSC from various studies for S/M testing.

D. **References:**

1. Drew S. Notebook No. 8950, pp.93-95 and Notebook No. 9057, pp. 1-25, 29.
2. Zoller, M. Notebook No. 9005, pp. 94-100 and Notebook No. 9045, pp. 1-15.
3. Hassam, S. Notebook No. 8823, pp. 95-103.

II. SUPPORT FUNCTION: SAMPLE PREPARATION

- A. **Objective:** To prepare chemical fractions and/or condensates as needed for biological and chemical analysis.

- B. **Results:** A sample of tobacco hornworm purge fluid was separated using a 5 kD hollow fiber membrane. Both the concentrate and the filtrate were freeze dried and are to be tested by Project 1101 personnel.

Triplicate smokings of 2R1 cigarettes collected on Cambridge filters, in impaction traps, in impaction traps followed by processing and in Elmenhorst cold traps (-78°C) followed by processing are being collected for varied chemical analysis.

Hand made cigarettes (182) were prepared for Project 6908.

About 450 samples from various sources were assayed for nicotine.

C. References:

1. Izac, R. R. Notebook No. 8874, p. 90.
2. Hellams, R. Notebook No. 8959.
3. McGee, N. Notebook No. 9016, pp. 53-69.

PROJECT NUMBER : 8101
PROJECT TITLE : Cigarette Testing Services
WRITTEN BY : Joseph M. Garman
PERIOD COVERED : February, 1991

I. MARKET ACTIVITY

- A. **Objective:** To monitor and report new brand introductions and brand modifications for the domestic and international cigarette markets.

B. **Results:**

Domestic

Philip Morris is marketing Marlboro 83 (Box) in Fargo and Duluth. The filter length increased by 2 mm (19 to 21 mm). This product delivers 16 mg/cigt. tar and 1.0 mg/cigt. nicotine.

International

Stingray King Size cigarettes were introduced in Japan in January by Japan Tobacco, Inc. This product delivers 11 mg tar and 0.9 mg nicotine. Stingray has a dual carbon on CA/CA filter. Seven rows of micromechanical perforation yield 28% ventilation. An unusual characteristic of Stingray is the presence of citrate in the cigarette paper. All Japan Tobacco, Inc. products previously tested used fumarate as the burn component in the cigarette paper. Deliveries of 10 mg tar and 0.9 mg nicotine are printed on the pack.

American Tobacco Co. began exporting American Super Lights King Size (Box) cigarettes to Japan in December. This 83 mm product has dual carbon on CA/CA filter with 33% ventilation. It delivers 11 mg tar and 0.9 mg nicotine. The soft pack version (85 mm cigarette length) of this product was introduced in Japan in 1988. Deliveries of 9 mg and 0.8 mg nicotine are printed on the box.

In December, American Tobacco Co. began exporting American Lights King Size (Box) cigarettes to Japan. This 83 mm product delivers 13 mg tar and 1.0 mg nicotine. American Lights has a dual carbon on CA/CA filter with 16% ventilation. Deliveries of 12 mg tar and 1.1 mg nicotine are printed on the box. The soft pack version (85 mm cigarette length) of this product was introduced in Japan in 1987.

First Mild King Size cigarettes, manufactured by The London Tobacco Co., England were introduced to the Japanese market in August. This 84 mm product delivers 13 mg tar and 0.7 mg nicotine. First Mild has a dual paper/CA filter (plug-space-plug) containing carbon and silicate granules. The filter is ventilated (11%). Deliveries of 11 mg tar and 0.8 mg nicotine are printed on the box.

San Chi Gold King Size cigarettes are manufactured by the Kumning Cigarette Factory, China, and were introduced in Japan in October. This 84 mm product delivers 14 mg tar and 0.6 mg nicotine. San Chi Gold has a dual carbon on

paper/CA filter with no ventilation. The blend contains no reconstituted tobacco, expanded stems or expanded tobacco. Deliveries of 12 mg tar and 0.7 mg nicotine are printed on the box.

- C. Plans: Continue to monitor cigarettes introduced in the marketplace.

II. FTC NICOTINE/WATER ANALYSIS

- A. Objective: For standardization and to improve statistical validity, the analysis of FTC nicotine and water will be converted from two pads per analysis to one pad per analysis.
- B. Results: In order to accommodate the increased sample volume as a result of this procedural change, two additional Hewlett-Packard 5890 gas chromatographs and two additional Gilson dispensing/transfer stations have been ordered.
- C. Plans: This procedural change is targeted for July, 1991.

III. SMOKING MACHINE EVALUATION

- A. Objective: To evaluate smoking machine puff profiles.
- B. Results: Filtrona has been contracted to perform puff profile analysis on individual ports of all smoking machines in the Division. This work is now in progress and will take approximately one week to complete. Thus far, some problems such as timing and gear slippage have been identified.
- C. Plans: To include profile analysis as a regular feature of smoking machine maintenance. Discussions are on-going with QA regarding the purchase of a profile analyzer.

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